

***Too many pressure vessel codes?***

**W**HY is it impossible, in this era of scientific internationalisation, to standardise once and for all the numerous design codes for pressure vessels? There are few, if any, scientific principles underlying many of these national codes, yet when examining them dispassionately one could easily deduce that a pressure vessel constructed in one country was a completely different piece of equipment to a similar vessel constructed in another often neighbouring country.

Recently, whilst discussing the International Symposium on 'Pressure Vessel Research Towards Better Design' held last January, we pointed this out. Since then the subject has been raised by the British Welding Research Association.

The annual B.W.R.A. report for 1960 states that all research on welded pressure vessels is directed towards improved design. Although it is common finding of research and operating experience that present design is excessively conservative, exploitation of the strength reserve in carbon-steel plate has not been possible due to the need to conform to design codes. In fact, only in the nuclear power industry, where refinements in detailed design are made as a precautionary measure, has the modern knowledge of stress systems and material behaviour been put to use.

A programme of tests on low-alloy steel pressure vessels is currently in hand, to assess the strength advantages of such steels in pressure vessel service. It must be remembered that, under existing codes of practice, the greater yield strength of low-alloy steels cannot be fully exploited because design stress happens to be based on tensile strength. If the design stress were based on yield strength, the resistance to deformation for a given degree of over-pressure would be more uniform to mild and low-alloy steels so that the latter could be used with economic advantage. There is a strong suspicion, according to the B.W.R.A. report, that the resistance of stronger materials to repeated stress applications would be no greater than that of mild steel. It has been shown that, because of its greater yield strength, a low-alloy steel pressure vessel should suffer a smaller strain range than one in mild steel, hence its fatigue strength would be better.

The admission that in the U.K. the greater yield strength of some alloys cannot be exploited, is all the more strange when noting that in Sweden the yield stress has been used as criterion for strength for the last 25 years (this is discussed by E. Jonnerby, a leading Swedish design engineer in a special article this month on 'Pressure Vessel Design in Sweden').

There is no doubt that this bolder approach is quite compatible with a high degree of safety (safety

factors of 1.5 are used in Sweden for calculating wall thickness, and 1.1 for calculating flanges and heads). Can it be therefore that, due to this lack of standardisation and over-conservative approach, British plant constructors may lose substantial business to those countries which are prepared to apply techniques learnt from nuclear pressure vessel experience?

***Converting active waste to solids***

**A** QUESTION which we have often discussed in this journal is, what will be the magnitude of waste disposal once economic nuclear power has been achieved. In the United States it is estimated that approximately 65 to 70 million gal. of high-level waste are at present in storage at various sites. The U.S. nuclear waste process industry is expected to generate 36 million gal. waste by 1980, containing approximately 10 billion curies of fission product activity.

Due to the many disadvantages of storing liquid wastes in tanks, such as potential leakage and the necessity of liquid waste transfer for centuries to come, much development work has been directed towards designing practical alternative systems for disposal of these materials. In one method, suitable for processing fuels of the materials test reactor type, the acid aluminium nitrate waste from the processing of fuels may be converted to granular alumina by calcining in a fluidised bed at temperatures from 400° to 550°C. This process involves the injection of the waste solution through spray nozzles into a fluidised bed wherein the water evaporates, the nitric acid decomposes to water and oxides of nitrogen and the metallic nitrates decompose to the corresponding metallic oxides. Operating temperatures above 400°C. have resulted in a decrease in bulk density of the final solid product. The metallic oxides are retained in the fluidised bed and are removed as granular solids. This results in a six- to eight-fold reduction in volume and hence minimises tank storage failure.

A second promising method for calcining waste is currently being investigated at Hanford laboratories. This process consists of feeding liquid waste through a pneumatic nozzle into the top of a tower. The walls are heated to 850°C. by passing low-voltage current through the entire column, and water droplets pass down the tower through successive zones of evaporation, drying and calcination. Separation of solids from gases has been effected by either filtration or cyclone separators. (Although this method has not been previously applied to radioactive waste treatment, it has been developed in the pyrolysis of sulphite liquors at the Canadian Pulp and Paper Research Institute.) The solid product may be stored as a fine powder or may be converted to a glass product.

## ***Fine particle technology***

**I**MPROVEMENTS in fuel efficiency are generally attributed to improved boiler design and utilisation or careful selection of fuel to give a maximum quantity of steam raised per B.T.U. thermal input. These factors, important as they are, deal only with part of this very complex problem.

It is not sufficiently appreciated that overall fuel efficiency can be attained only if sufficient attention is also paid to fuel preparation prior to burning. In the case of coal for example, washing, screening, drying and grinding will determine the final quality of coal available for steam raising (and hence influence the fuel efficiency). These operations, which chemical engineers have long been carrying out in other chemical processes, are now receiving more attention by the coal industry than in previous years. The 1960 annual report of the British Coal Utilisation Research Association describes recent studies carried out on fine-particle technology.

A laboratory centrifugal coal pulverising machine has been tested in the B.C.U.R.A. laboratories and it was confirmed that its new design gives the expected flow of feed material into the grinding zone. Studies of crushing mechanisms have also been started using photo-elastic materials to represent both particles and grinding elements. By means of this technique the stress pattern can be observed visually as a system of fringes. On theoretical expectations, a stress pattern should be established in 10 microseconds or less and such patterns would be expected to change rapidly with alterations in the magnitude and pattern of the loading. High-speed photographs have shown that it is possible to determine the stress distribution, but a much shorter time interval between photographs than the present equipment limit of 130 microseconds is needed, if the changes are to be followed.

During work carried out on pulverising, it was found that sieving of ground coal, containing a large proportion of fines, can give spurious results. A series of tests was carried out to define a more satisfactory technique, since the errors are affected by the quantity and particle size of material being sieved.

## ***Industrialising Pakistan***

**W**HETHER Pakistan, with a population of 86 million and a *per capita* income of £25 per annum, will ever raise her standard of living depends not only on the success of the Indus project but also on the rate of industrialisation.

The second five-year plan, 1960-65, which the Pakistani government recently inaugurated, intends to base industrial development on existing raw materials—jute, cotton, sugar cane and natural gas. Minerals such as chrome, iron, bauxite, chalk, gypsum and rock salt have been found, but so far not exploited. Perhaps the most valuable underground asset is natural gas, which is available in abundant quantities in both West and East Pakistan. Already two pipelines have been built from the Sui gas fields to Karachi (a distance

of about 300 miles), where they serve as fuel for various industrial undertakings. So far, surprisingly, few oil deposits have been found—despite the fact that the Soviet Union has been intensively prospecting for oil and other minerals. Nevertheless, Pakistan has decided to have her own refinery with a capacity of 1.5 million tons p.a. Kellogg International Corp. is currently erecting this refinery and Bataafsche Internationale Petroleum Mij. will be in charge of technical supervision when the refinery comes on-stream in 1962. The Pakistan government has also given a contract to the Swiss consulting firm Imhausen to advise on the possibility of establishing a petrochemical industry to produce polyacrylonitrile, acetylene and PVC. Several plants already manufacture nitrogenous fertilisers; Union Chimique Belge recently built a 50,000-ton-p.a. ammonium sulphate plant and a French consortium, Schneider-Creuzot and Saint Gobain are erecting a 103,000-ton-p.a. ammonium nitrate plant and a 59,200-ton-p.a. urea plant. A Japanese company, Kobe Steel Works, is also building a 117,000-ton-p.a. urea plant which should be on-stream by the end of this year.

One great drawback to rapid development in the chemical industry is the dire shortage of basic chemicals such as caustic soda, chlorine and sulphuric acid. Although several heavy inorganic plants are in existence, many built with German technical aid, it is futile to plan an ambitious petrochemical, plastics and dyestuffs industry until these basic chemicals are produced in sufficient quantity—whether this can be done depends entirely on foreign capital investment and on an assured confidence in the financial and political stability of the country and its future.

## ***Steam wetness determination***

**S**TEAM from boilers often has a high percentage of water entrained with it. This water is responsible not only for corroding boiler tubes and pipelines, but also for lowering considerably the thermal value of steam. Although it would therefore be helpful if the quantity of moisture in steam could be checked continuously, there are several factors that make this very difficult to achieve with any degree of accuracy. Methods of determining the wetness fraction of steam were outlined in a paper recently presented at the Institute of Fuel by D. C. Gunn.

The author described the nature of steam-water mixture as it exists inside the pipe from which it is to be sampled. Under practical conditions, steam flow in a main is turbulent, with the result that water droplets soon reach the walls of the pipe where they merge and form a film surrounding a dry core of steam. To obtain a representative sample from this system is obviously a difficult matter, because it involves withdrawing samples both of steam from the core and water from the pipe which are proportional to their respective flows.

Some approaches to this complicated problem were suggested as follows:

(1) Foaming, the most frequent cause of heavy

carry-over, should be eliminated by addition to the boiler water of suitable anti-foam solutions.

- (2) The whole of the steam output of the boiler should be measured independently by means of a steam meter and the whole of the moisture in the steam offtake should be trapped in a calibrated steam separator and measured.

There are several available methods of simplifying wetness measurement. The first of these involves the use of apparatus for dispersing the liquid on the pipe walls into the steam. It has been shown that the liquid film adhering to the pipe wall is ruptured when the steam flow exceeds a critical value. Therefore, by accelerating the steam flow to several times its critical value, some of the surface film will be dispersed into the steam. Several 'dispersers' are, in fact, available at present.

Another method would be to drain the steam main by means of a pot, welded into the bottom of the main, and measure the water drained. Such a drain might only remove 20% of the water in the system, but this of course, is considerably better than nothing at all. A refinement of this method would be two or three such drains separated by several feet along the steam main.

### **Britain and Euratom**

ONE of the distinct advantages to be gained from Britain's entry into the Common Market would be her eligibility to join Euratom, the nuclear energy association of the European Economic Community. Much of Euratom's work consists of developing economic nuclear power reactors to satisfy the specialised needs of Western Europe—with abundant and readily available supplies of coal and oil. Although in this country the U.K.A.E.A. has, during the last decade, gathered much valuable experience in nuclear power, most of this has been exclusively in the rather specialised branch of gas-cooled graphite-moderated reactors. What will happen when the Calder Hall type of reactor has been developed as far as it can go, by about 1967? In this journal we have often drawn attention to this one-sided attitude of the British government to nuclear power (*C.P.E.*, 1960, p. 327). In fact, there might well be a serious crisis in the British nuclear power industry by the end of this decade unless alternative nuclear power reactors are developed using enriched rather than natural uranium.

The financing of Euratom is obviously a costly business. At present France and Germany, the biggest contributors, have each paid £64.5 million during the last five years. The Euratom budget for research during this period has been £76.5 million and it is expected that this amount will be doubled for the second five-year period beginning in 1963. Due to the many imponderable factors in reactor development it will be impossible to forecast precisely which types will prove most suitable in 10 years time—but by then Euratom should have gathered sufficient experience to help the entire European Economic Community.

### **Expenditure in the oil industry**

THE oil industry is an international undertaking, cutting right across strictly national frontiers. Besides being ubiquitous, it is also engaged in practically every aspect of the chemical industry as well as such important service industries as transportation, construction and storage. It is perhaps a good example of the benefits of internationalisation.

This can best be appreciated by an examination of recently issued figures which relate the growing toll of capital expenditure to which the oil companies have been committed. Between 1950 and 1959 inclusive, a total of £30,000 million was spent in the Western world alone on exploration, the expansion of production facilities, and the replacement and development of property, plant and equipment of all kinds. The overwhelming proportion of it had to be found by the oil companies themselves, through lack of any alternative source of funds on the requisite scale.

The level of expenditure required has steadily risen year by year, so that in 1959 almost two and a half times as much was spent as in 1950. It is also interesting to observe that a growing proportion of capital is going to countries outside the U.S. (whereas from 1950 to 1955, the U.S. usually accounted for approximately two-thirds of total expenditure, by 1959 its share had fallen to slightly less than half).

Year by year, exploration and production receive the major share of oil-industry expenditure, and over the past decade this item has accounted for more than 58% of the total. The next most expensive sector has been refinery construction, with over 14%.

Tankers, marine equipment and marketing facilities together absorb most of the oil industry's remaining capital outlay. One relatively small, but rapidly increasing, item is the construction of chemical plants. This averaged 1.4% of total outlay between 1950 and 1959, but in the last year the proportion stood at 3.1%—clear evidence of the oil industry's growing interest in the petrochemical industry.

### **Cracking waxy distillates**

INDIA is faced by a steadily increasing consumption of petroleum products. The consumption pattern, however, is such that the demand for middle distillates in the form of kerosene and diesel oils is much higher than for gasoline. (This is broadly the trend in Europe and for this reason European refineries differ so significantly from American refineries.)

In a paper presented at the symposium on 'Hydrocarbon Synthesis and Conversion', held in Bangalore recently, A. N. Narayanaswamy and others discussed the refining of recently discovered Indian crudes, in order to obtain the maximum amount of middle distillates. This particularly referred to the processing of heavy distillates and residues from the crudes discovered in the Assam province. As part of this investigation, the work on the catalytic cracking of waxy fractions (boiling point, 350° to 400°C.) over synthetic silica-alumina catalyst was reported. An experimental laboratory plant was designed consisting



of a constant-rate liquid feeder, reactor, condenser system and constant-pressure receiver. The upper part of the reactor was filled with glass beads and catalyst was packed below this. Fractions boiling at 350° to 400°C. were obtained by vacuum distillation and used as feedstock; experimental runs were made to investigate the effect of temperature, space velocity and duration of cracking, keeping the other process variables constant in each case.

In these experiments runs were made at a liquid hourly space velocity of 0.5 for 90 min., it was seen that the optimum yield of middle distillate (43%) was obtained at 400°C. Furthermore, it could be clearly noticed that the yield of gasoline and middle distillates gradually rises up to a space velocity of 0.5, beyond which there is a decrease—on the other hand, the yield of gas decreases continuously with increase of space velocity. It is well known that gases from catalytic cracking generally are rich in  $C_3$  and  $C_4$  hydrocarbons because of the tendency of large fragments to crack. In this work it was also noted that concentration of  $C_3$  and  $C_4$  hydrocarbons in the gas exceeded the  $C_1$  and  $C_2$  concentration. However, conversion decreases as the duration of the run is increased due to progressive coke deposition on the catalyst.

Unfortunately, the authors reported that the middle distillate obtained did not come up to the diesel index specification for grade A or B. They suggest, therefore, that hydro-cracking over a suitable catalyst would be more successful.

### **Accident rate in mines**

THE Chief Inspectorate of Mines and Quarries, established by Parliament under the Mines and Quarries Act, 1954, performs a valuable service to all those in industry who are vitally concerned with the prevention of accidents. The 1960 report of the Chief Inspectorate, which has just appeared, makes stark reading. A table of general statistics from 1853 to 1960 shows that output of coal reached a peak of 260 million tons in the period 1913-22. In 1960, output was considerably less at 188 million tons. The number of persons annually killed in mines was 1,012 from 1853-62, 877 from 1933-42 and 317 in 1960. This is certainly an improvement; however, the accident rates show just the opposite trend. The total number of injuries per thousand persons employed was 148 from 1903-12, 181 from 1933-47 and 306 in 1960. During the last two years particularly, this rate has gone up alarmingly.

It is pointed out in the report that last year was marred by what is described as the worst explosion in South Wales, when 45 persons were killed. An outstanding feature of many explosions in mines, was the large numbers which were caused by frictional sparking during orthodox coal-cutting operations. The main factor causing these ignitions is the presence near the cutting edge of a band of hard material or of intrusions of quartzitic sandstone. Ignitions of fire-damp in a machine undercut are always difficult to suppress, since the work exposes the fire fighters to

considerable dangers because of the possibility of ignition of larger accumulations of firedamp. Thus, unless the fire can be put out quickly, it may be necessary to seal off the district and to reopen it later—a very dangerous operation.

The risk of extensive coal-dust explosions has been increased by the development of modern methods of working and transporting coal. The report on the South Wales Six Bells colliery explosions mentioned that the limitations of general stone-dusting on conveyor roads, led some years ago to the conclusion that an additional safeguard was necessary and that stone-dust barriers should be used for this purpose. Although they cannot prevent an ignition of coal dust, they can limit the extent of an explosion.

It is obvious that many explanations can be given to interpret these alarming statistics. Yet the sombre fact remains that an increasing accident rate in coal mines is hardly likely to improve the 'prestige' of already expensive coal.

### **Boiling-water reactor**

EUROPE'S first boiling-water reactor has now been in continuous operation for over a year at Halden, southern Norway. The reactor, which is a joint project between O.E.E.C. and the Norwegian Institute of Atomic Energy, was designed to take into account Norway's unique energy reserves. Due to her cheap supplies of hydro-electricity, Norway was one of the first countries in the world to produce heavy water on an industrial scale (it may be recalled that Norwegian heavy-water plants proved of immense value during the second world war). It was quite natural, therefore, that any Norwegian power reactor system would have to be based on heavy water. Furthermore, joint agreements made after the war between the Norwegian and Dutch governments assured Norway of supplies of enriched uranium at favourable prices. Hence the decision to build a boiling-water power reactor, fuelled by either enriched or natural uranium using heavy water as modifier and cooler.

A recent progress report by the Kjeller Research Establishment states that, for the first quarter of this year, the Halden reactor has been in more or less continuous operation to perform power experiments with the first fuel charge. Temperature coefficient measurements at temperatures up to 150°C. have been performed, together with water level coefficient measurements, using natural uranium fuel elements. In addition, a xenon poisoning experiment has been performed to determine the reactivity of xenon.

On the engineering side, the main emphasis has been placed on the planning and preparation of modifications that must be done to the plant for operation with the second charge. A new steam transformer is to be installed and operated in parallel with the original one. The existing secondary side will be made into a closed loop in order to obtain better control with the feed water and to eliminate danger of stress corrosion. A tertiary loop with a new steam generator will also be installed.



# Pressure Vessel Design in Sweden

By E. Jonnerby,\* Dipl.Ing.

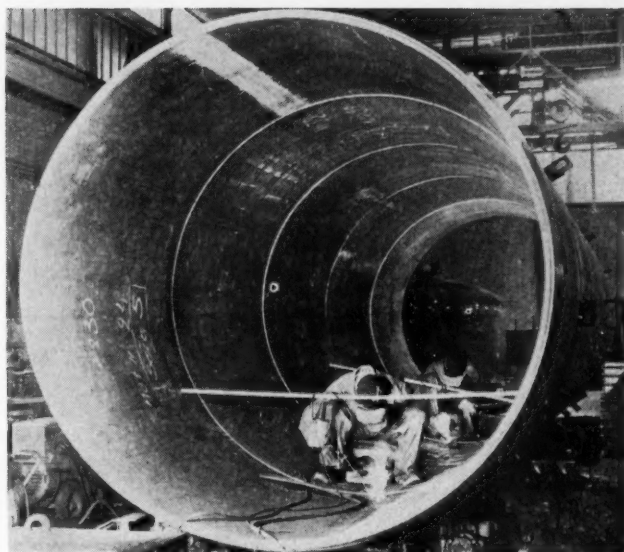


Fig. 1. Manual arc welding of the cylindrical shell of a solid stainless-steel reactor at a design pressure of 1,450 p.s.i.

*Sweden has long been renowned for the high-quality steels produced locally. As a result of this, a substantial plant fabricating industry has been established to serve the growing chemical and nuclear industries. In this article Swedish pressure vessel codes are discussed and compared with their British equivalents. Due to Swedish designers working on the yield strength rather than tensile strength, different safety factors and design pressures are used.*

WHEN designing pressure vessels nowadays, the designer has a very long list of specified types of steel, but in extreme conditions it is often the quality of steel that decides on its technical possibilities.

The precondition for industrialisation is that, besides having a sufficient amount of material of suitable quality, it is necessary to obtain energy in a suitable form and at a low cost. Hitherto, steam has been the largest source of power. However, its future is questionable, taking into consideration the supply of coal and oil, but for some time to come the stocks of these materials will be sufficient. In producing energy from such sources, and also from nuclear energy processes now being developed, there is always some sort of pressure vessel that forms the basic object of design. In pace with the efforts to mass-produce energy at a low cost, the dimensions of pressure vessels are steadily increasing and often reach the limits of present-day techniques. The chemical industry also demands larger units and in the 'container'

age in which we live, considerable material and numerous vessels for storing and transporting of chemicals, oils, liquefied petroleum, etc., are needed. In connection with the manufacture of these large-sized and 'dangerous' vessels, the methods of fabrication have also changed, especially welding, which has become the most prominent technique.

From a security point of view it is vital to draw up codes for the estimation and fabrication of such plant which could cause serious accidents through failure.

Material research has provided an answer to the technical requirements for harder steels and better endurance against corrosion and brittle fracture. In the first place, there has been a conspicuous development towards high-strength steels, i.e. steels with higher yield point, higher tensile strength and higher impact strength at temperatures of about 0°C. The higher strength is of great importance as regards the wall thickness and the weight of boiler drums and similar vessels, as well as autoclaves and re-

actors for the chemical industry. The transportation problems play a large and decisive part today and the handling in the shop is also directly dependent on the weight of each plant. Fabricated plant components of 100 tons and more are becoming increasingly common nowadays.

The demand for high impact strength at low temperatures is of importance for vessels which contain liquefied gases at low temperatures. Even if there is no direct correlation between the frequency of brittle fractures and impact test values, it is important that correct procedures governing material composition and preheating of the material during pressing and welding are observed and used.

Corrosion resistance as well as post-welding heat treatment are often specified, and extensive research has been carried out in these fields. There are, however, other factors which are equally important—the strength of the

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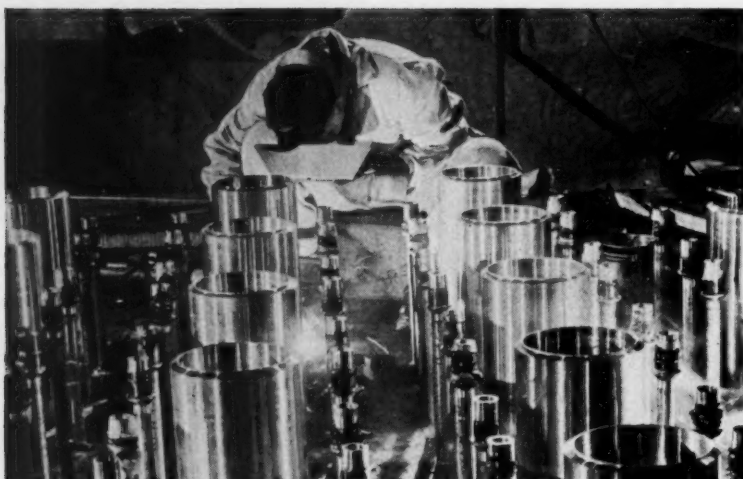


Fig. 2. Different tubes for fuel element charging and discharging passing through the heavy walled cover of the reactor

material and the weldability.

The strength of the material can be increased for example, by cladding mild steel with stainless steel, but even this operation requires highly skilled technicians when fabricating high-pressure reactors, autoclaves, heat exchangers, etc.

### Safety factor

For nearly 25 years Sweden has used the yield point as the criterion of strength for specifying the wall thickness of pressure vessels. According to the Swedish pressure vessels code, a calculated value is specified for each standardised material just a little below the yield point. Depending upon the type of the stress, the degree of safety has been determined. The maximum allowable stress value for a material is obtained by dividing its calculated value by a safety factor which for mild steels is 1.5 at pure membrane stresses and 1.1 for bending stresses. A safety factor of 1.5 is consequently used when calculating the wall thicknesses of cylindrical and spherical shells, while a safety factor of 1.1 is used when calculating flanges and heads. The following basic formulae for calculating the plate thickness of thin-walled vessels subjected to internal pressure are given as examples.

#### (1) Cylindrical shells:

$$S_{min} = \frac{Dp}{200 \times (\sigma/S_F) \times Z}$$

where

$S_{min}$  = minimum thickness in mm. of the plane plate for manufacture

- $D$  = inner diameter of the shell, mm.  
 $p$  = calculated pressure (design pressure) in atm.  
 $\sigma$  = calculated value for the yield point in kg./sq.mm.  
 $S_F$  = safety factor  
 $Z$  = efficiency of joints, etc.

#### (2) Non-stayed dished ends:

$$S_{min} = \frac{D_y p y}{200 \times (\sigma/S_F) \times Z} + b$$

where  $S_{min}$ ,  $p$ ,  $\sigma$ ,  $S_F$  and  $Z$  are as before, and

- $D_y$  = outer diameter of end plate in mm.  
 $y$  = stress concentration factor depending on shape of end plate  
 $b$  = 1.5 mm. (usually) = an additional value intended, *inter alia*, to compensate for the thinning of the material produced in manufacture.

Depending on the material quality, such a calculation gives a divergent stress-level in service, as compared with the British and American codes. If the ratio between yield point and tensile strength is high, the material, according to Swedish codes, will have higher stresses and the dimensions will be smaller than if the maximum allowable stress value is determined as one-quarter of the tensile strength. When the yield point : tensile strength ratio is low, which is the case with the stainless austenitic steels, the relationship will be the contrary. For the more common steels at reasonable temperatures, the Swedish code specifies lower thicknesses as compared to

calculations according to the British codes. The energy content which is stored between yield point and tensile strength justifies such a procedure. No accident or failure due to this type of calculation has been reported.

As will be seen above, the Swedish formulae in principle correspond to British ones, but the determination of safety factor and design pressure may vary. According to Swedish codes the joint efficiency of the weld can range through 0.6, 0.75 and 0.9 depending on the intensity of control—particularly the radiography—and the heat treatment to which the vessel has been subjected. As a lower limit of thickness of material that must be heat-treated,  $\frac{5}{8}$  in. is valid. However, in using the modern pressure vessel steels with favourable transition temperature, this limit can be increased, after approval by the Swedish Authority for Pressure Vessels.

### Test pressure

The test pressure for mild steel vessels in Sweden has been determined to be 30% above the working pressure. If a higher test pressure is required, the design pressure has to be altered accordingly. Vessels of stainless steel or of stainless-steel-clad plate are tested at a pressure equivalent to



Fig. 3. The first commercial Swedish nuclear reactor vessel under completion at the Degerfors Works of Uddeholm Co., Sweden

the working pressure and, of course, the design pressure must be the same. This does not correspond to the rules of Great Britain and U.S.A. The codes in these two countries specify that the actual strength of materials measured on every sheet may be used, and that these actual material values determine the maximum allowable stresses. The calculated values of the materials indicated in Swedish handbooks and codes have been specified below the minimum yield point, giving a statistical safety factor.

When designing structures with modern high-strength steels, more comprehensive calculations must be carried out than those previously used. The lower dimensions used demand special control of the stability characteristics bearing in mind the fact that stability and buckling are primarily dependent on the modulus of elasticity and the moment of inertia, and only secondarily on the tensile strength. Therefore, it is necessary to pay special attention to stability characteristics when using high-strength steels. Oscillating phenomena that sometimes arise must be taken into consideration; for large storage tanks, pulp digesters, etc., a number of oscillations lower than three per second cannot generally be accepted. Below this number the oscillating motion can be caused by manpower, and the amplitude becomes critical. Above this number the oscillation is noticed either as a trembling of the vessel or as a tone-like sound.

Modern boiler drums have operating pressures up to 2,000 p.s.i. and temperatures up to 750°F. For such purposes low-alloy steels with high yield strength are preferred in Sweden, particularly molybdenum-boron steel with high yield strength compared with the best mild steel, has secured a good market during the last few years. When using such a steel in preference to the best mild steel, the wall thickness of certain vessels can be reduced by 50% according to the Swedish codes.

Vessels made in such a high-strength steel may be more elastically deformed than those made in conventional steels, and the pure elastic elongation in the material at working pressure may amount to approximately 0.10%. This means for example, a considerably higher swelling of the tube holes in boiler drums and heat exchangers. Therefore, the tubing must be carried out more efficiently than normally. The design of the tube holes, arrangement of brackets and rings must be done with due consideration for the large elastic deformation.

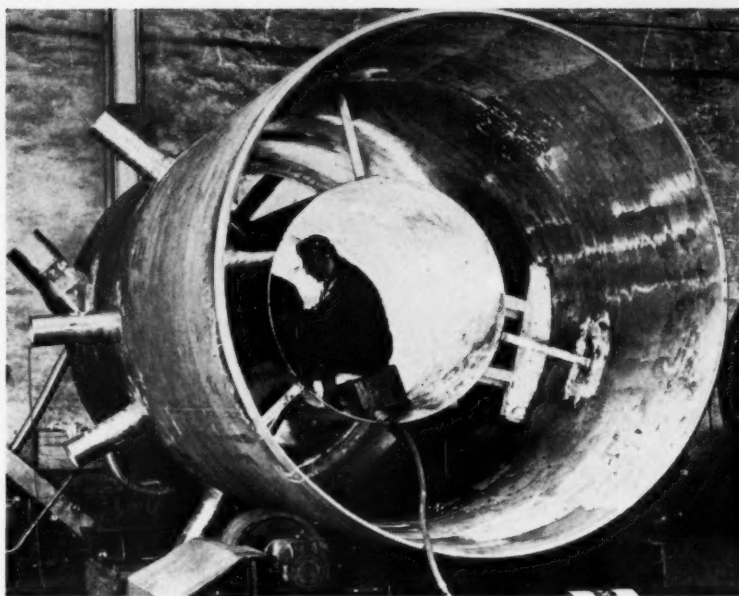


Fig. 4. A clad-steel pressure vessel with a design pressure of 570 p.s.i. under fabrication

#### Radiographic examination

According to the Swedish code for pressure vessels, there are three classes of radiographic examination:

- (1) Complete radiographic examination of every weld.
- (2) Radiograph A.
- (3) Radiograph B.

The complete radiographic examination is prescribed for pressure vessels which, owing to contents and size, are considered particularly dangerous (e.g. vessels for town gas, hydrogen gas, liquefied petroleum gases and chlorine). Radiograph A comprises radiographic examination of all longitudinal welded joints for the whole of their length, including points of intersection. Radiograph B comprises radiographic examination of at least 10% of the total length of the welded joints according to Radiograph A. It also includes all joints at intersections and the joints subjected to the severest stresses.

High-pressure vessels require more rigorous supervision in the fabricating shop, and the extension of radiographic examination may amount to 300% for heavy-walled high-pressure vessels.

In the first instance the root passes are controlled. The next step is examination after welding about half the thickness, and the final radiographic examination is made after completing the welds. At thicknesses over 3 in., ultrasonic testing is used in addition to radiography; the tendency

is to use more double or triple examination. The surfaces are often examined by supersonic or magnetic particle equipment as well as by dye penetrants, the latter being specially used for the examination of overlay welding of stainless-steel overlaying of heavy flanges and plates which cannot be fabricated from sandwich-rolled clad steel plate.

#### Nuclear reactors

Heavy-walled reactors for chemical and nuclear plants are being built in Sweden. One example is the reactor vessel of Sweden's first commercial nuclear reactor built by the Degerfors works of the Uddeholm Co. This reactor vessel has an inner diameter of 15 ft. and the walls are 2½ in. thick of stainless-steel clad plate. The design pressure is 570 p.s.i. and the design temperature 250°C. The weight of the reactor pressure vessel, with cover, loose flanges and bolts, exceeds 170 tons, and the total weight including stainless-steel internal components is about 320 tons. Figs. 1, 2, 3 and 4 give an idea of the dimensions of this reactor pressure vessel.

The design of a still larger reactor vessel for the next Swedish nuclear project has advanced so far that basic data are already available. The design pressure will be 820 p.s.i., the inner diameter of the reactor vessel 17 ft., the wall thickness up to 7 in., and the giant pressure vessel will have a weight of very nearly 300 tons, excluding the cover and all internal fittings.



# Development and Planning of Chemical Processes

By Prof. Dr. K. Schoenemann\*

*Due to the rapid growth of chemical production throughout the world, new potential processes must be investigated and developed more quickly than before, and the number of scale-up steps reduced. In this article, examples are given in which chemical engineering techniques, particularly kinetics and reactor design, are applied to two processes: phenol synthesis from benzene and furfural production from xylose. Both processes have been closely studied by the author at Darmstadt, and illustrate the present-day importance of optimisation.*

**P**RESENT-DAY methods of developing and designing chemical processes are determined by the rapid rate of technical progress. This is reflected by the growth of world chemical production, bearing in mind a fact reported by many leading chemical companies that some 40% of their turnover consists of products which have only appeared on the market during the last ten years. The change in trend experienced since World War 2 points to a structural change based on the fact that the growing consumer demands of the rapidly increasing world population can no longer be met without the aid of chemistry. Production is no longer, as in former days, based mainly on auxiliary chemicals. Nowadays it is mainly based on catalytic methods, usually in the

form of continuous processes undergoing extreme reaction conditions, requiring complicated plant equipment.

Because of the rapid rate of technical progress, new methods must nowadays be developed up to full production scale in much less time than previously, when it was customary to progressively enlarge existing equipment. Whereas in former years a reaction process was treated rather intuitively, during the last 20 years there has been a continuous trend towards determining quantitatively the different reaction factors by means of controlled experiments leading towards precise calculations. As a result, it is now possible and without risk to scale-up processes in a single step. In general there is no longer any need for semi-technical pilot plants except

for the solution of quite specific problems or for the advance production of adequate quantities for market research.

For such quantitative advance planning, the Anglo-Saxon countries have created a new and highly specialised scientific discipline known as chemical engineering, which now has as many students as the three classical chemical disciplines taken together. In Germany the teaching of this discipline, known as chemical technology, is virtually confined to the technical universities, where it is regarded as the fourth pillar of chemical education. This conception will be useful as long as the field does not become too wide.

In view of the vast field which comprises chemical technology, the dual task of surveying methods applicable to the development and planning of chemical processes and of characterising this new discipline are here carried out with the aid of two typical examples. In order to do this, subject matter contained in 200 to 300 university lectures has been condensed into this article.

## Raschig's phenol synthesis

The increasing demand for phenol, especially in plastics, can no longer be met using coal tar as raw material; as a result, a number of synthetic processes have been developed on the basis of the more readily available benzene (Fig. 1). Despite the high

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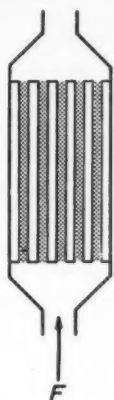
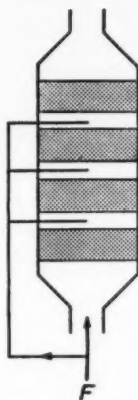
Process	Reactions	Chemicals	Product	Yield
		kg	kg	%
			ref to benzene	
Sulphonation	 $\xrightarrow{\text{H}_2\text{SO}_4}$  $\xrightarrow{\text{NaOH}}$ 	90 $\text{C}_6\text{H}_6$ 160 $\text{H}_2\text{SO}_4$ 155 NaOH	100 Phenol	85-92
Dow-Process	 $\xrightarrow{\text{Cl}_2}$  $\xrightarrow{\text{NaOH}}$ 	95 $\text{C}_6\text{H}_6$ 85 $\text{Cl}_2$ 125 NaOH	100 Phenol	80-90
Raschig-Process	 $\xrightarrow[\text{Air}]{\text{HCl}}$  $\xrightarrow{\text{H}_2\text{O}}$ 	100 $\text{C}_6\text{H}_6$ 17 HCl 220 Air	100 Phenol	75-85
Cumene-Process	 $\xrightarrow{\text{C}_2\text{H}_6}$  $\xrightarrow[\text{Air}]{\text{CH}_3\text{CH}_2\text{CH}_3}$  + $\text{CH}_3\text{COCH}_3$	95 $\text{C}_6\text{H}_6$ 50 $\text{C}_2\text{H}_6$ 140 Air	100 Phenol 35 Acetone	80-90
Direct Oxidation	 $\xrightarrow{\text{O}_2}$ 	160 $\text{C}_6\text{H}_6$ 730 Air	100 Phenol	30-60

Fig. 1. Industrial phenol processes

### Multitube Reactor



### Multibed Reactor



### Fluidized-bed Reactor

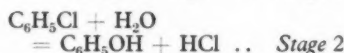
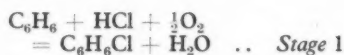


Fig. 2. Types of catalytic gas-phase reactors

consumption of chemical intermediates, the conventional sulphonation process is still economic, thanks to measures taken which have improved its economic efficiency.

Liquid-phase chlorination followed by hydrolysis under pressure of the chlorobenzene with sodium hydroxide, also entails a high consumption of chemical intermediates. Its economics depend entirely on the price of chlorine. With the Raschig method, chlorination is carried out catalytically using hydrochloric acid and air, and the hydrochloric acid is subsequently recovered during hydrolysis. The economics of this process are governed decisively by the higher capital costs of the catalytic processes. The Cumene process, which likewise works without chemical intermediates, will only be economic if a market can be found for the by-product, acetone. Direct oxidation of benzene into phenol is still in the early stages of development.

To demonstrate the interdependence of chemical processes and chemical engineering design, the Raschig process will be described, since this gives the best results. The overall equations are as follows:



The process may be regarded as an oxidation of benzene by means of air.

### Types of catalytic reactors

The reactor is the heart of the process, because the type and size of the separation units are determined by the composition of the product mixture

which consists of the desired main product, undesired by-products and unconverted raw material.

During the first stage, benzene chlorination, the decisive factor is the lowering of the reaction temperature, by means of the catalyst, to such an extent that benzene combustion is suppressed. It is also necessary to use a large excess of benzene so that higher chlorination is suppressed by the mass effect and all the hydrochloric acid is entirely consumed, if possible, since its recovery from a dilute solution is uneconomic. Moreover, the large excess of benzene acts as heat carrier, thus facilitating temperature control. Experiments have shown that a 10-fold molar excess of benzene and a 2.5-fold molar excess of air are desirable.

For a highly exothermic reaction of this kind, it is in principle possible to use one of the three following types of reactors (Fig. 2):

- (1) The multitube reactor offers a large heat-transfer surface for the coolant but has a relatively small catalyst charge.
- (2) The multibed reactor is cheaper because of its simple design. Reaction heat is absorbed after each bed by the entry of cold or evaporating base material.
- (3) The fluidized-bed reactor is isothermal; due to the effective intermixing obtained by the whirling catalyst pellets, the reaction heat can be removed by cooling units.

When designing a reactor, it is not enough to determine from semi-technical trials (Fig. 3) the final conversion from this and the space-time yield, because the temperature profile arising due to heat formation and heat dissipation does not necessarily apply to other reactors. It is, in fact, necessary to obtain more exact data concerning the reaction rate as a function of temperature and concentration.

### Determination of reaction rate

The reaction rate can only be determined experimentally. In the case of catalytic processes, there are, in principle, three available possibilities.

(1) In the integral reactor (Fig. 4) a feed  $F$  is converted by a large catalyst charge of weight  $W$ , to a fairly high percentage  $X$ . If it is possible to keep the process isothermal (e.g. if heat of reaction is low), then, for a given temperature and different time factors, points on a reaction/residence time curve are obtained. In this connection, it is advisable to express the residence time by the time factor, i.e. the ratio of the catalyst weight  $W$  to the hourly throughput  $F$ . In other words, the greater the catalyst charge for a given throughput, the longer the

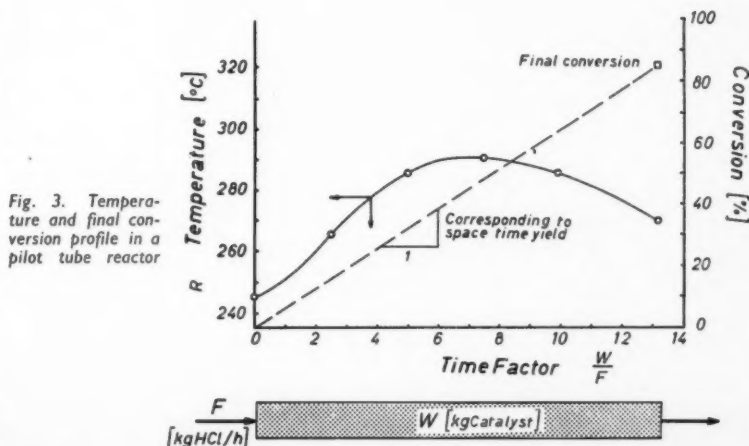


Fig. 3. Temperature and final conversion profile in a pilot tube reactor

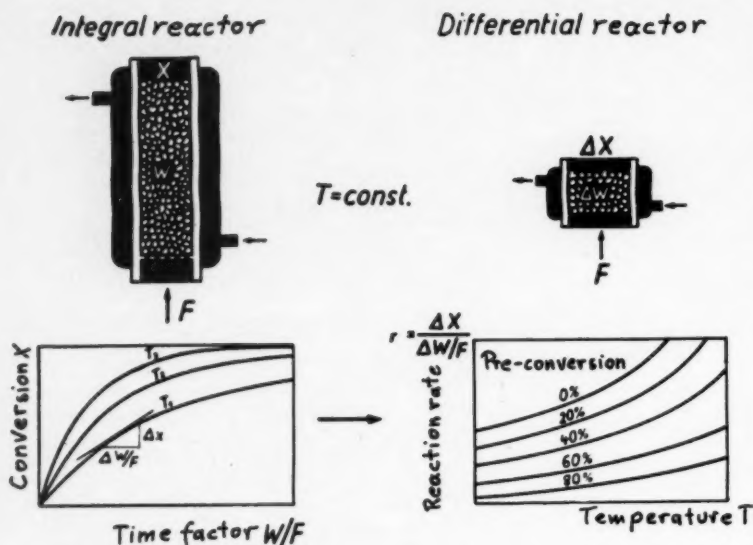


Fig. 4. Possibilities of experimental determination of reaction rates

residence time. Differentiation of the reaction curve with respect to the time factor gives the reaction rate  $r = dX/d(W/F)$  as a function of conversion. The reaction rates thus obtained, if plotted against the reaction temperature, give the reaction rate diagram (Fig. 4, bottom right).

(2) If it is not possible to make the process isothermal, the reaction rate can be determined directly from the very short contact layer of the differential reactor where no considerable temperature rise can take place. But the analytical determination of the minor changes in concentration encountered in this process is often difficult.

(3) A more elegant way of determining the reaction rate than by means of a differential reactor is the use of an integral converter in which an axial temperature profile is formed whilst the radial profile can be neglected (Fig. 5). In this case, not only the temperature profile, but also the course of the reaction is determined by taking samples at different contact levels (or else by a series of experiments having equal throughput with catalyst charges at different heights). By differentiating the S-shaped reaction curves thus obtained, the reaction rates for given pre-conversion are obtained. These rates are now plotted, point by point, against corresponding temperatures (e.g. in the steps of 10% as shown in Fig. 6) in the form of a reaction rate/temperature diagram. The curves, here shown in heavy lines, indicate the reaction rate as a function of the reactor length. Points with equal pre-conversions can then be linked to form

the curves of the complete reaction rate diagram.

The apparatus used for benzene chlorination (Fig. 7) was operated with different heights of catalyst bed. The results are shown in Fig. 5.

#### Reactor calculation

The reaction rate diagram, thus obtained, can then be used to solve the specific problem of calculating the shape and size of the reactor. The multitube reactor is found to be most suitable for the example here discussed.

The top line of Fig. 9 defines the reaction rate  $r$  described above, as a function of the conversion  $X$  and the temperature  $T$ , the dimensions being moles of reaction product per kilogramme of catalyst per hour. The calculation of the reactor amounts to

determining, for a certain feed  $F$  and a given conversion  $X$ , the requisite catalyst weight  $W$ . The solution of this equation for the ratio  $W/F$  which, as already explained, represents a modified retention time, yields the second equation in Fig. 8.

The integral on the right-hand side can be solved in one of two following ways:

(1) As long as it is possible for given reaction rates to formulate a reaction rate equation in the well-known manner, the integral can be solved for an isothermal process. In the case of a non-isothermal process, the integral can be solved numerically by introducing the heat balance [ $T = T(X)$ ]; this can easily be done with the aid of computers.

(2) If, on the other hand, the reaction mechanism is so complicated that a reaction rate equation cannot be formulated, it is also possible to use the reaction rate diagram ( $r/T$  diagram) referred to above for the calculation of  $W/F$ . Starting from a preliminary conversion of  $X = 0$  at the inlet temperature  $T$ , the process may either be isothermal, where the entire reaction heat is removed by a suitable cooling system, or adiabatic, where no heat is removed at all. In practice, one generally works between these two extreme cases. In our case, the reaction rate diagram must be plotted step by step from the heat balance: heat of reaction = self-heating + heat removal, until the desired final conversion  $X$  is reached. The reciprocal values of the intermediate reaction rates  $r = f(T)$  are then plotted against the conversion  $X$ . The area under the curve thus obtained, ranging from  $X = 0$  to  $X = X_{final}$ , gives the required value  $W/F$  from which, for a given

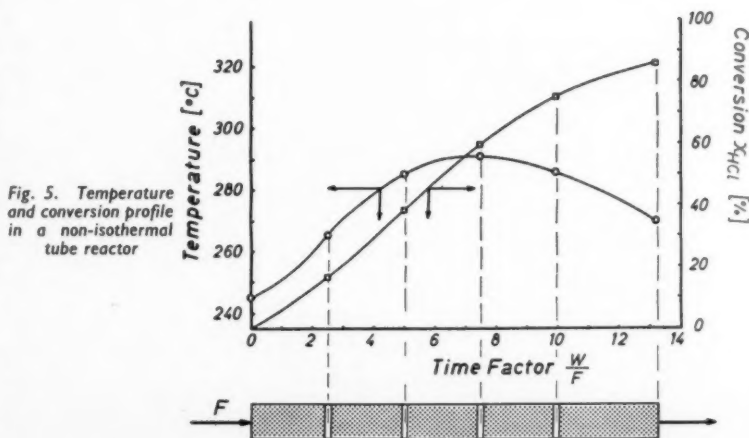


Fig. 5. Temperature and conversion profile in a non-isothermal tube reactor



throughput  $F$ , the necessary catalyst weight  $W$  can be calculated.

Both methods of calculation, i.e. the numerical and graphical method, are based on the simplifying assumption, which is often incorrect in practice, that there is no radial temperature gradient in the catalyst bed. In fact, the temperature generally drops in the direction of the cooling walls. In this case, the cross-section of the reactor is divided into concentric rings and the temperature and conversion diagrams are calculated for these. With the benzene chlorination process, 97% hydrochloric acid must be used up, since, in view of the high degree of dilution, recovery is not economic.

The reactor for the chlorobenzene hydrolysis is similarly calculated.

### Separating units

The small degree of chemical conversion obtained with benzene chlorination as well as with chlorobenzene hydrolysis, gives rise to substantial recycling, which governs the whole

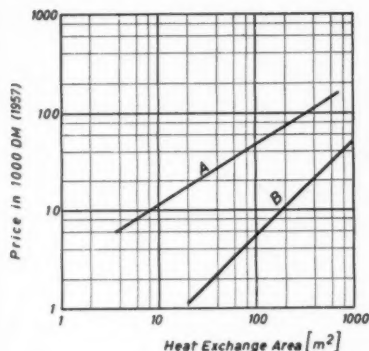


Fig. 8. Costs of heat exchangers. A: multi-tube exchangers. B: air-cooled exchangers (from Koelbel-Schulze)

aspect of the process. In designing the individual stages, many other considerations must still be taken into account and certain measures to improve the economic efficiency must be taken which, for brevity's sake, are only briefly mentioned.

Benzene, dilute hydrochloric acid and air enter the chlorination reactor in the vapour state. The chlorobenzene formed is condensed, separated by distillation and then hydrolysed with water in the vapour phase. This process is limited to 10% conversion in order to avoid undesired side reactions. From the reaction mixture which remains in the vapour state, hydrochloric acid is removed by absorption with water and is recycled to the chlorination reactor. The phenol is subsequently absorbed in water at a

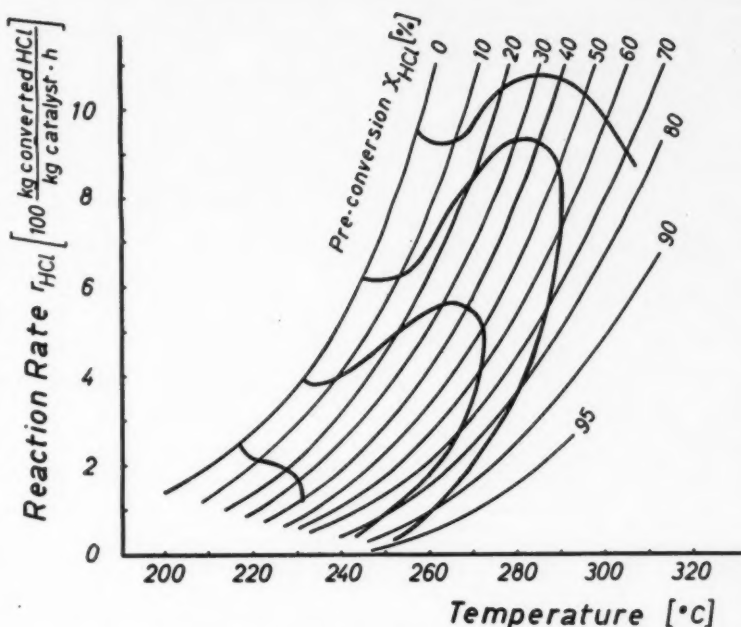


Fig. 6. Diagram of the rate of chlorination of benzene (at 10-fold excess of benzene and 2.5-fold excess of air)

slightly lower temperature and is extracted from the aqueous solution with benzene. The benzene is evaporated and recycled to the chlorination process, while the phenol remains as a residue and is purified by distillation.

In this way, apparatus required for every stage of the process is designed specifically as regards shape and principal dimensions. Energy consumption

(steam, electricity, cooling water) is calculated and the capital cost estimated (Fig. 8). The detail design as governed by mechanical strength and economic construction is a matter for the engineer.

### Layout of plant

In order to determine the most suitable layout, taking into account simplicity of piping, possibility of

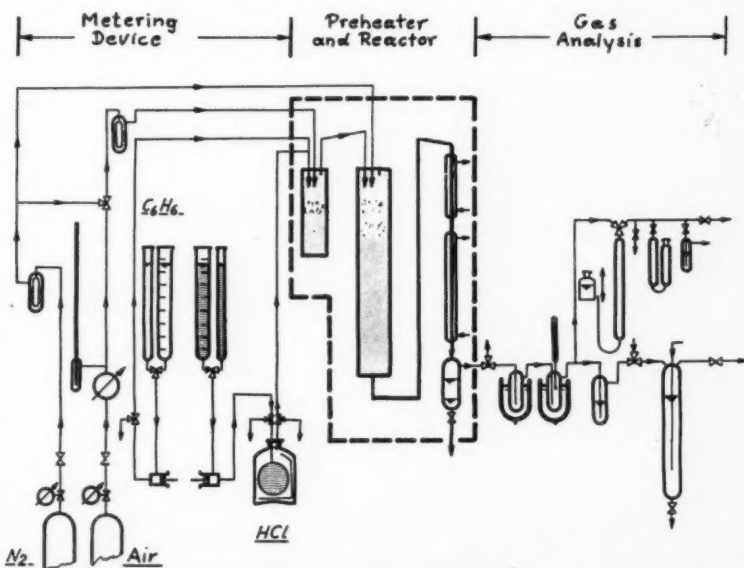


Fig. 7. Apparatus used for benzene chlorination

group supervision and control, fire risk, etc., it is advisable to build a model of the entire plant. A scale of 1 in 100 or 1 in 50, for a capacity of 6,000 tons of phenol p.a. is usually selected.

The capital cost of the plant, works out at about 10 million DM.

In calculating the expected production costs (Table 1), it is necessary, for comparative purposes, to adopt a common scale of DM./100 kg. of phenol produced, for all different cost items such as raw materials, energy consumption, wages, repairs, amortisation and interest. Only then will it be possible to compare the proposed process with alternative processes and to estimate the decisive advantages of the process. For example, in this case, raw materials represent 45%, and the cost of energy 20% of the total production cost. In spite of the numerous plant, the depreciation, amounting to 13% of the production costs, is not a significant factor, the annual production being valued at 6,000 tons  $\times$  1,160 DM./ton = 7 million DM. The capital cost amounts to the 1.5-fold of the annual production value which is, strictly speaking, in excess of the level considered as tolerable for modern catalytic synthetic processes.

### Furfural synthesis

So far this article has dealt with the design and planning of a catalytic gas-phase reaction process. It is now intended to stress the same approach, illustrated by another example, the production of furfural from aqueous xylose solution, a by-product of the selective hydrolysis of the carbohydrates of wood.

The process consists of the cleavage of three molecules of water by means of acid catalysis (Fig. 10), performed in an homogeneous liquid phase where the difficulties associated with temperature control are eliminated. The basic factor governing the design of the process is, as before, the operation of the reactor; the reactor calculation must again be based on a knowledge of the reaction kinetics. Xylose reacts in accordance with a first-order reaction (dotted curve in Fig. 11). Yet the furfural yield is far from the theoretically expected value because furfural is further converted to a resin. This latter process is also of first order. If a main reaction of first order is followed by a consecutive reaction also of first order, the yield should be independent of the initial xylose concentration. But, as has been proved by experiments with dif-

$$\text{Determination of the Reaction Rate } r(X,T) = \frac{\partial X}{\partial W/F} \left[ \frac{100 \text{ kmol R}}{\text{kg catalyst hour}} \right]$$

$$\text{Determination of the Time Factor } \frac{W}{F} = \int_0^X \frac{dX}{r(X,T)} \left[ \frac{\text{kg catalyst}}{\text{kmol A/hour}} \right]$$

Numerical Integration  
of a Rate Equation

$$r(X,T) = k(T) \cdot (1-X), T = T(X)$$

$$\frac{W}{F} = \int_0^X \frac{dX}{k(T) \cdot (1-X)} \left[ \frac{\text{kg catalyst}}{\text{kmol A/hour}} \right]$$

Graphical Integration  
of a Rate Diagram

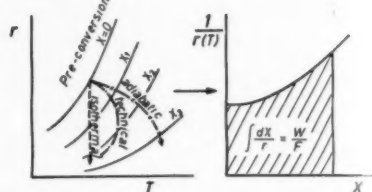


Fig. 9. Principles of the calculation of a reactor for a catalytic reaction  $A \rightarrow R$

ferent initial concentrations, this is not the case.

This phenomenon can be interpreted only by assuming a more complicated reaction mechanism shown below, according to which the furfural formed is condensed with a partially dehydrated intermediate product. From this, the reaction rate equations quoted

are derived. The reaction rate constant of the xylose decrease  $k_1$ , and of the resin formation  $k_3$ , have been determined experimentally. The condensation reaction cannot be obtained experimentally. The furfural formation equation, essential to the calculation of the reactor, combines these three equations as shown in Fig. 12.

Table 1. Expected production costs of phenol in Germany

German Marks per 100 kg. phenol based on prices of 1959

Capacity 6,000 tons/year

	Quantity per 100 kg. phenol	Price per unit (DM.)	Cost per 100 kg. phenol (DM.)
(A) RAW MATERIALS:			
Benzene, pure .. ..	103 kg.	50	51.50
Hydrochloric acid .. ..	45 kg.	10	4.50
Catalyst .. ..	2 kg.	200	4.00
Total (A) .. ..			60.00
(B) CREDIT FOR BY-PRODUCTS ..	8 kg.	100	8.00
(A) minus (B) .. ..			52.00
(C) POWER:			
Steam .. ..	0.9 tons	15	13.50
Electricity .. ..	100 kWh.	0.05	5.00
Water .. ..	50 cu.m.	0.04	2.00
Gas .. ..	150 N cu.m.	0.02	3.00
Total (C) .. ..			23.50
(D) OPERATING LABOUR, 1 man-hr. ..		3	3.00
(E) SUPERVISION, 30% of (D) ..			0.90
(F) MAINTENANCE:			
6% per year of 8 million DM. apparatus investment .. ..			8.00
2% per year of 2 million DM. building investment .. ..			0.70
Total (F) .. ..			8.70
(G) SUPPLIES, 15% of (F) .. ..			1.30
(H) GENERAL SERVICES:			
60% of (D) + (E) + (F) + (G) ..			8.30
(I) AMORTISATION:			
10% of apparatus investment ..			13.30
5% of building investment ..			1.70
Total (I) .. ..			15.00
(K) TAXES, INSURANCE, LICENCE:			
2% per year of total investment ..			3.30
Production cost, 100 kg. phenol			DM. 116.00

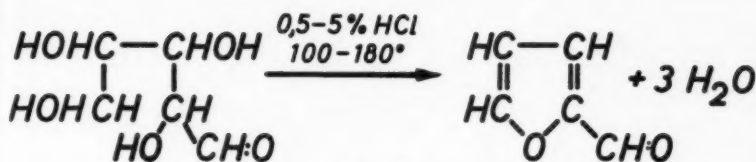


Fig. 10

The real furfural formation is therefore equal to the theoretical formation calculated from the decrease in xylose, less the furfural losses through condensation and through resin formation. From this relationship, it is possible to work out conversely the reaction rate constant  $k_2$  of the condensation reaction from the real formation rate of the furfural.

This kinetic model has since been confirmed under different conditions of continuous operation, viz. with the tubular reactor and with the stirred tank reactor in which, because of the back-mixing, quite different concentration conditions are encountered. Fig. 13 shows the excellent accordance of the theoretical conversion curves with the data for different residence times obtained experimentally. But, due to the rapid side reactions, the furfural yield of about 8% of the theoretical value is extremely low.

If the reaction rate equation of furfural formation is reviewed once more with a view to suppressing the furfural-consuming side reactions, the only possible way appears to be to keep the furfural concentration in the reaction mixture as low as possible. This is also the basis of the conventional methods of producing furfural from vegetable material rich in pentosan, where the furfural thus formed is continually removed by steam. With

pure aqueous xylose solution, however, the extraction of furfural by means of a high-boiling solvent, e.g. tetralin, is a more economic proposition.

The quantity of furfural then remaining in the aqueous phase, which continues to be exposed to the undesirable side reactions, is the lower the higher the partition coefficient and the greater the quantity of solvent, i.e. the greater the ratio of solvent to aqueous phase,  $q_S/q_R$ . The intimate mixing of the two phases calls for the use of agitated vessels. Within each of the two groups of curves (Fig. 14), the difference between co-current and counter-current flow, or between different numbers of agitated vessels, is not great. The higher yield of the upper curves, amounting to over 60% of the theoretical value compared with 45% of the lower group, is due to the higher solvent to raffinate ratio. The pre-calculated values were corroborated by the results of a pilot plant (Fig. 15).

These considerations have formed the basis for calculating and designing a production plant, in a similar way as in the phenol synthesis discussed previously. The economic optimum is represented by a cascade of three vessels with co-current flow of xylose solution and tetralin (Fig. 16). From the separated tetralin solution, furfural

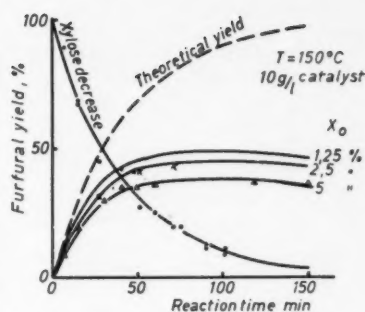


Fig. 11. Theoretical and effective furfural yield and xylose decrease

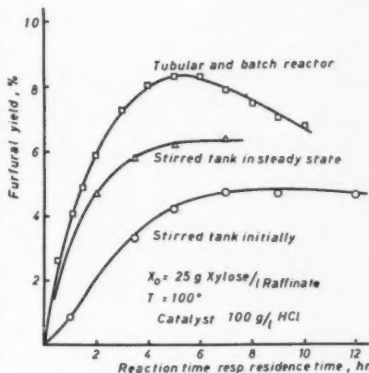


Fig. 13. Experimental check of the calculated furfural yield in tubular and batch reactor, stirred-tank initially and stirred-tank in steady state

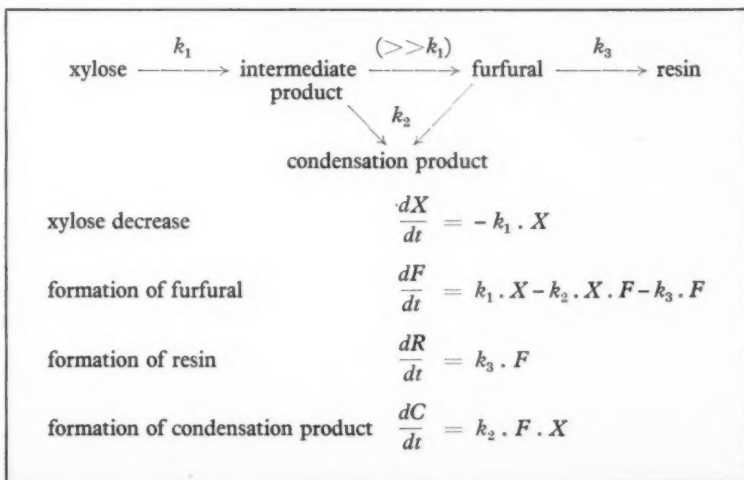


Fig. 12

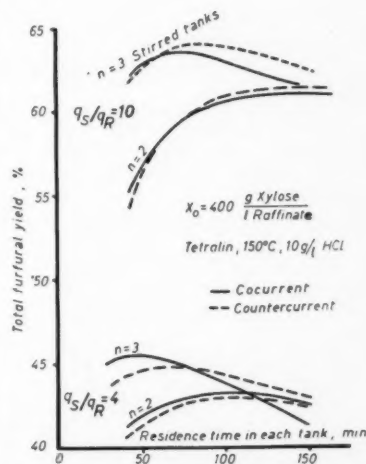


Fig. 14. Total furfural yield with co-current and counter-current extraction, for different ratios of solvent to aqueous phase



is distilled off as the top product in a vacuum distillation column and then further rectified. The hot tetralin is recycled so that the large quantity of solvent merely requires inexpensive pumping.

### Conclusions

In view of the marked optima of furfural yield, it is easily appreciated that the economic optimum, too, can be found by the aid of computers with an interlinked system of equations in which the different cost items are included—a modern method known as optimisation. Furfural synthesis is a good example illustrating another topical trend—the increase of selectivity, i.e. the promotion of the desired reaction, which in this case is

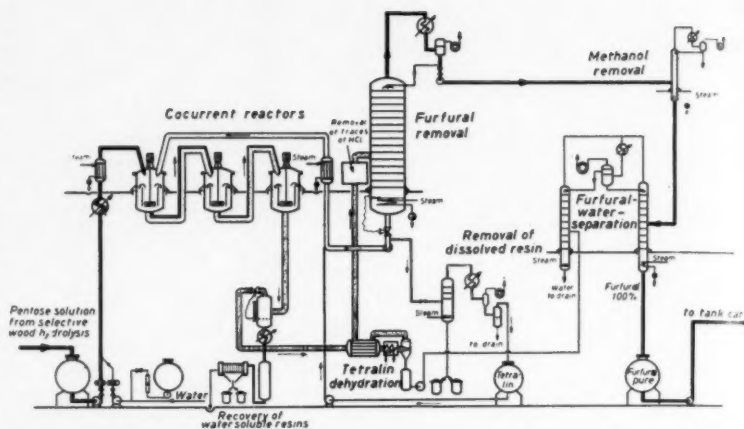


Fig. 16. Preliminary flowsheet for the production of 300 tons/month furfural

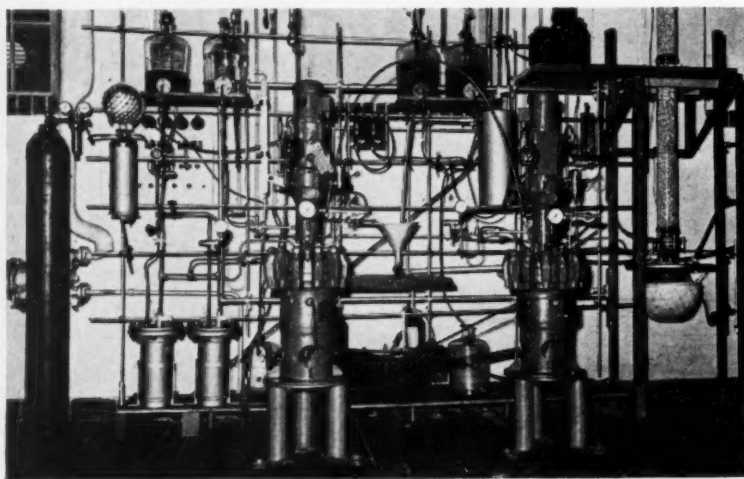


Fig. 15. Pilot plant for production of furfural

obtained by the extraction of the product.

The modern trend is towards far-reaching automation where it is essential to adjust the characteristics of the control equipment to the characteristics of the reactor. In this connection, reactor stability is of major importance inasmuch as, for example, the heat dissipation must be adjusted to the heat formation in a way that ensures a reliable control under all reaction conditions.

At Darmstadt, the basic knowledge required for such plant design provides the subject-matter for the training in chemical engineering for all students of chemistry. Such plant design, correctly carried out in this way, is the most comprehensive and logical system to be found in all the manifold aspects of the chemical industry.

## INDUSTRIAL PUBLICATIONS

**Heat-transfer medium.** In an illustrated brochure, published by Hygrotherm Engineering Ltd., brief descriptions of chemical plant design are given and *Hygrotherm* systems of operation explained. Other sections deal with indirect heat transfer, heat generators and mains frequency induction heating, with a list of properties and useful conversion factors to conclude.

**Nitrogen and hydrogen plants.** The Incandescent Heat Co. Ltd. have published a new bulletin (No. V68) describing their range of hydrogen and nitrogen plants and the processes involved. Flow diagrams are included and there are summaries of operating costs and utility requirements.

**Gas scrubbers.** These are the subject of an illustrated booklet published by Chemico-Chemical Construction (G.B.) Ltd. The operating principles and performance data of the venturi scrubber type P-A and S-F and the p-A cyclonic scrubber are given.

**Heat-transfer medium.** The Heavy Organic Chemicals Division of I.C.I. have issued a leaflet on *Thermal*. Various charts illustrate its physical properties and suggestions are made as to its general applications.

**Rubber.** A new method of increasing the flame resistance of natural rubber is described in the Natural Rubber Bureau's quarterly journal entitled *Rubber Developments*.

**'Terylene'.** A new I.C.I. illustrated booklet considers some of the parts which *Terylene* plays as an industrial fibre and includes general information on the properties of *Terylene* relating to industrial applications.

**Contactors and extractors.** An illustrated brochure (P-100) describing the Podbielniak line of centrifugal contactors, solvent extractors, chemical reactors, separators and clarifiers is available from Clark Bros. Co. Division, Dresser (G.B.) Ltd. The operating principle of these machines is described and there is a comprehensive section on applications. The brochure concludes with specifications for the full line of Podbielniak contactors and separators.

# MIXING

By G. J. Jameson,\* B.Sc., A.S.T.C.

*Despite the importance of the mixing process, it is still one of the most empirical unit operations in technology. This annual review surveys development in the theory of mixing and novelties in design of mixing plant for liquid-liquid, liquid-solid and solid-solid phases. Mixing of non-Newtonian fluids is discussed and the influence of back-mixing in continuous flow systems is mentioned. Altogether 93 references are made to the literature and to patents.*

MIXING is one of the oldest of industrial arts, yet even today, in spite of the giant strides being taken by man in more sophisticated branches of technology, it remains a mainly empirical process. From the technologist's point of view, the obstacles to basic research have been formidable, as there was until recently no means of measuring the degree of mixing achieved by a given process except by rule-of-thumb methods peculiar to the process or industry. A notable contribution was made by Danckwerts<sup>1</sup> when he defined several mixing criteria which enabled the performance of actual mixers to be assessed quantitatively, giving a solid basis for further research. Also, there was no general explanation for the many manifestations of the mixing process. A significant advance was made when Lacey,<sup>2</sup> Danckwerts<sup>1</sup> and others proposed that all mixing took place by one or more of the following mechanisms: (i) convective mixing, transfer of clumps of matter from one location to another; (ii) diffusion, distribution of particles over a freshly developed surface; and (iii) shear mixing, setting up of slip planes in the mass.

Another restraining influence on the investigation of the mixing process was the lack of real incentive to increase the efficiency of industrial mixers. In many processes, mixing is rather an incidental, comparatively cheap operation and, if the mixer was not efficient, it merely meant that it must be run for a longer time. Where mixing is the main objective, however, the capital cost of equipment can be considerable, and poor design and operation become costly. With good design and operation under optimum conditions, it is obvious that there will

be many cases where savings can be made without sacrificing quality. This need of industry for more efficient mixing is reflected in the increasingly quantitative approach being made to the subject and, although the most rapid steps have been taken in the study of liquid mixing, some notable advances in dry solids mixing and the mixing of non-Newtonian fluids are reported in this review.

## Mixing in liquids

The literature shows that more effort is being made to apply basic engineering principles to this aspect of mixing. A detailed analysis of the hydrodynamic behaviour of liquids during mixing with radial paddles and turbines has been attempted,<sup>3,4</sup> giving equations of flow of particles in the upflow and downflow regions and in the turbulent region surrounding the mixer shaft. An approximate mathematical model for the mixing process in a stirred tank is proposed by Marr,<sup>5</sup> who showed that the liquid level has a critical influence on the performance of the tank.

Ruckenstein and Smigelschi<sup>6</sup> determined an eddy diffusivity for mixing in a stirred reaction vessel and compared it with that obtained in a fixed bed and in counter-current flow. Using the results of experiments on various types of propeller and turbine stirrers, Lienerth<sup>7</sup> has designed a new type of stirrer with curved paddles which is more efficient than the turbine type, especially at low speeds.

By assuming that close to the turbine mixer rotating in a baffled tank there is a small volume of perfectly mixed fluid surrounded by unmixed fluid, Norwood and Metzner<sup>8</sup> derived a model of the mixing process in a

baffled tank for the laminar and early transitional periods.

The visualisation of flow patterns continues to be of interest. Following on the papers that have appeared in recent years describing the use of fluorescent particles and radioactive tracers, Metzner and Taylor<sup>9</sup> report the use of small tracer particles in highly illuminated narrow beams of light. A further improvement on this method, devised by van Meel and Vermig,<sup>10</sup> consists of dividing the flow field into a number of highly illuminated light strata of different colours, so that three-dimensional visualisation is possible.

The anchor agitator for batch mixing can now be added to the number of impellers and agitators for which power consumption data are available. Uhl and Voznick<sup>11</sup> have constructed a plot of the power number as a function of Reynolds number, with clearance as a parameter.

A general discussion of the factors to be considered when designing fluid mixers was given by Dykman.<sup>12,13</sup> Corrosion control, a factor which is often overlooked in the design of mixers, was elaborated by Parker.<sup>14,15</sup>

For continuous mixing of two or more liquids, Molyneux<sup>16</sup> has described a simple venturi mixer which can be used as a combination jet mixer and entrainer. The drive of a high-speed agitator is often a source of trouble when dependable sealing is required.

Schulze-Bergkammen<sup>17</sup> has described a new type of drive which is claimed to be of small height, a reliable guide for the shaft and to offer depend-

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able sealing. Variations on more conventional apparatus have been described.<sup>18-22</sup>

The Cemac *Agidrive* has been developed to provide stirring of large vessels with reliable sealing between shaft and vessel.<sup>23</sup> The main feature is the long spacing of gearbox bearings which gives greater shaft rigidity for large unsupported lengths. It is claimed that shafts up to 25 ft. long can be used without a bottom steady bearing. Speed can be readily changed by means of pick-off gears, and the absence of external ribbing, allowing easy cleaning, is welcome.

A new type of mixer based on the effect of ultrasonic motion on liquids is the *Polytron*.<sup>24</sup> The system is said to represent a marked advance in homogenisation, emulsifying and dispersion technique. In addition to the turbulence set up, which is the same in action as in propeller-type mixers, energy is released at a much greater intensity by ultrasonic cavitation, giving a large increase in mixing efficiency so that process time is greatly reduced and product quality improved. The high-intensity ultrasonic vibrations needed for this type of mixer were formerly generated by electronic means, both expensive and comparatively inflexible in operation. The *Polytron* uses a new system which is little more complex than the conventional high-speed rotary mixer, and

which generates vibrations of up to the order of megacycles/sec. Consistencies of fluids mixed may vary from coarse particle or fibre suspensions down to low-viscosity Newtonian fluids. The relative simplicity of the mixer head permits a wide range of models, ranging from a micro-apparatus for processing volumes of the order of a few centimetres of liquid up to production installations of 200 or more gallons. Fig. 1 shows an ultrasonic homogeniser made by Ultrasonics Ltd. Q.F.V. Ltd. also make a mixer, similar in operation to the *Polytron*, and known as the *Vibro mixer*. Mixers with capacities from 2 to 600 gal. are available.

The disadvantages of pipeline mixers which have retarded their use in industry are that the pressure drop over such mixers is generally high, and the length of mixing tube needed for reasonably efficient mixing is often considerable. The *Suma* pipeline mixer made by the Sugar Manufacturers Supply Co. has been developed to overcome both of these difficulties. The principle of operation is that the main flow of fluid is split into a number of smaller streams after passing over a guide or distributor cone, and made to flow in contra-rotating spiral streams around the periphery of a tube. The unit may be placed in an existing flow line and the mixing takes place *en route* through it. If one of the streams to be mixed is considerably smaller than the major flow, it can be introduced on to the distributor cone by means of a small pipe in the assembly. This type of mixer is most suitable for gas and low-viscosity liquids and suspensions mixing. Among suggested uses are boiler water treatment, blending of petroleum fractions, treatment of effluent and gas-liquid contacting.

The *Pioneer* portable mixer<sup>26</sup> uses conventional propeller agitators and is now available in an improved type of mounting. Similar types of mixers are also made by Premier Colloid Mills Ltd. Their *Premier* range of laboratory, pilot-plant and production models can be supplied with either marine-type propellers or the patented *Dispersator*, which is especially recommended where high shear is necessary, and is satisfactory with viscosities up to 5,000 cp. Premier also produce a range of robust side-entry mixing units with power inputs up to 15 h.p.

Plenty & Son Ltd. manufacture a range of liquid blending equipment, but their speciality is a mixing head known as the *Impelator* (see Fig. 2). Tubes placed at an angle on the peri-



Fig. 1. Ultrasonic homogeniser made by Ultrasonics Ltd.

phery of a circular plate provide mixing in place of the propeller.

### Mixing of solids

Experiments on simple forms of mixer with binary mixtures continue. Yano,<sup>27</sup> having studied mixing of dry powders in the double-cone, cube and horizontal cylinder<sup>28</sup> and the effects of physical properties of solid powders<sup>29</sup> in recent years, has continued his work with an investigation of mixing in a ribbon mixer. His overall mixing curves are similar to those obtained with rotational mixers, but the influence of rotational speed on the number of revolutions required to produce a given standard of mixing is less than in the rotational type. This is in line with the rather different mixing mechanisms involved—convection in the ribbon type compared with mainly diffusional mixing in the rotational type. Yano reports that the optimum volume ratio of charged powder to volume of mixer is 17.3%. Gayle and Gary,<sup>30</sup> who also seem to have embarked on a long-term study of solids mixing, have reported on mixing rates in horizontal drum-type mixers. Their results seem to indicate that dry solids mixing can be better described by a second-order rate equation than by the first-order equation formerly assumed.

Weydanz<sup>31</sup> has used methods evolved by Coulson and Maitra<sup>32</sup> to investigate the course of the mixing process as a function of time (with regard to rotating horizontal drums). The equations developed are used to check the efficiency of a newly developed tetrahedral rotating mixer which is shown to be highly effective.

A general review of the design of solids mixers and blenders is given by Rathmell.<sup>33</sup> Design and performance criteria are compared for vertical and horizontal mixers. General improvements to existing patterns continue to be made.<sup>34-37</sup> A new multi-stage mixer of the dry-pan type common in the ceramic industry is reported.<sup>38</sup>

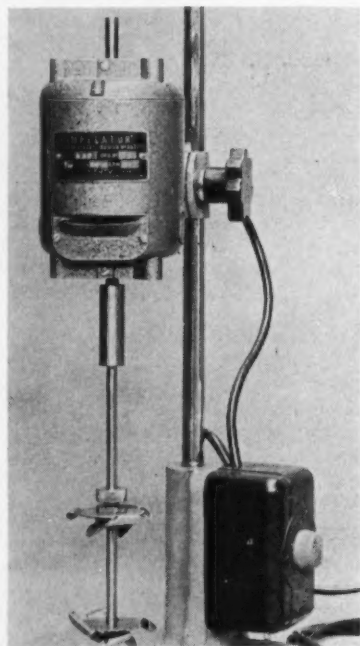


Fig. 2. Mixer with 'Impelator' head made by Plenty & Son Ltd.



Mixing of solids is generally thought of as a batch process, but a new process is described for the continuous blending of dry materials.<sup>39</sup> The Patterson-Kelley Co. Inc. have added a combined blender and dryer to their already large range of conical, vee and ribbon mixers.<sup>40</sup> Known as the solids processor, it is based on the Patterson-Kelley type of rotating vee mixer (the *Twin-Shell*) and it will, in sequence, dry blend, disperse liquids throughout, granulate and dry, solid powders. A method this company uses to pre-test and scale up to customers' requirements has been described,<sup>41</sup> and an article written by an engineer of the company has compared various mixers, with special reference to the *Twin-Shell* type.<sup>42</sup>

A mixer similar to the tetrahedron described by Weydanz<sup>21</sup> is the subject of a recent patent.<sup>43</sup> A further form of tumbler mixer is the *Rotocube*, in which a cube-shaped drum rotates at a compound angle. The motion of the drum imposes a figure-of-eight mixing action on the powder, so that mixing is particularly effective, and powders of widely differing densities and particle sizes may be handled. When the ingredients have a tendency to agglomerate and ball up, impellers which rotate slowly in the direction of rotation of the drum can be incorporated and undue impacting of the materials is avoided. The *Rotocube* is made by Foster, Yates & Thom Ltd.

Apex Constructions Ltd. now market a range of granulating mixers intended for blending of tablet masses.<sup>44</sup> The mixers are of the trough type with longitudinal agitators of various patterns. It is interesting to note that the manufacturers recommend that the working capacity of these mixers is the swept volume of the mixer blades, which is considerably more than the figure of 17.3% of the mixer volume reported by Yano<sup>27</sup> above. It is well to remember, however, that the optimum volume for a given standard of mixing is not necessarily the optimum economic volume.

The technique of fluidisation is the basis of a new powder mixer manufactured under licence by Gardners of Gloucester. Air blasts through the mixing head at the base of the machine agitate the powder in a spiral action which is claimed to give a perfect mix ten times faster than conventional mixing methods. Liquids can also be mixed with the powder as required. The main advantage is that there are no moving parts in contact with the powder, so wear and contamination are minimised.



Fig. 3. Manesty '300' stainless-steel mixer made by Manesty Machines Ltd.

A new glassed-steel conical dryer-blender is announced by Pfaudlers.<sup>25</sup> It is similar in action to conical tumbler mixers and dryers already available from a number of manufacturers, but its special construction permits contact with any acid except hydrofluoric and with alkalis up to pH 12 at 212°F. The inner tank can withstand full vacuum or up to 20 p.s.i., and the jacket can accommodate pressures up to 40 p.s.i. The equipment is marketed by Enamel Products Corp. (1933) Ltd.

#### Liquid-liquid mixing

Main interest in this section is devoted to the formation of disperse phases of high surface area. In a new approach to the analysis of dispersions in stirred tanks, Shinnar and Church<sup>45</sup> demonstrated that the concepts of local isotropy developed by Kolmogoroff can be used to describe the behaviour of turbulent flow in stirred tanks and for predicting particle sizes in liquid-liquid dispersions. Hills<sup>46</sup> has derived expressions for the scale-up of agitation systems for emulsions and suspension polymerisations. The resulting equations can only provide a rough guide to the order of the variable to be expected in a scaled-up plant, as important factors such as impeller design and the position of the impeller in the tank are not considered. No experimental results are given.

McDonough *et al.*<sup>47</sup> have studied the formation of interfacial area in an orifice mixer and derived a satisfactory correlation, using the well-tried photoelectric method to measure the surface area.

The stabilisation of dispersions, such as occur in suspension polymerisations, by turbulence, has been studied by

Church and Shinnar.<sup>48</sup> Equations are derived which, for a given agitation system, describe the region of droplet stability on the droplet diameter-stirrer speed diagram. Apparatus for mixing and homogenising liquid-liquid dispersions have been reported,<sup>49, 50</sup> the latter being based on a centrifugal principle.

An exact solution for the laminar mixing of two contacting semi-infinite parallel streams of gas flowing at constant velocity but at differing temperatures has been proposed.<sup>51</sup> The results are compared with the simple solution obtained by assuming a Lewis number of unity.

#### Solid-liquid mixing

Slurries have recently been used in an increasing number of industrial applications, and interest is also being shown in the use of slurries in nuclear reactors. Weisman and Efferding<sup>52</sup> have studied the behaviour of an aqueous thorium-uranium slurry, and their results are in agreement with the results for different systems obtained by Kneule<sup>53</sup> and Zwietering.<sup>54</sup> Their work is mainly in the turbulent region, and more work in the laminar and transitional zones is needed.

Mixers for suspending clay in water have been described,<sup>55, 56</sup> the latter making use of a perforated wall to keep large clumps of unbroken clay near the impeller. The cyclone is now used for mixing solids with fluids.<sup>57</sup>

For intimate mixing of powders or moist masses the Manesty '300' mixer has been designed (see Fig. 3). Made in stainless steel, the sturdy paddle driven by gear drive makes this mixer suitable for handling highly viscous materials.

## Reaction vessels, mixer-settlers

Following a recent paper<sup>58</sup> on the determination of mixing efficiency for continuous flow systems, in which mathematical models were proposed to represent various mixing conditions, Cholette *et al.*<sup>59</sup> have investigated the performance of flow reactors at various levels of mixing between the two extremes of the tubular and the continuous-stirred tank reactor. This analysis, although confined to rather simple models, should lead to a better understanding of the performance of flow reactors in general. In a gas-phase flow reactor stirred by the entering feed jets, Bartok *et al.*<sup>60</sup> have studied the overall mixing with a krypton-85 tracer.

The effect of back-mixing on conversion in chemical reactors was recently reported by Levenspiel and Bischoff.<sup>61</sup> Szekely<sup>62</sup> pointed out some defects in this work, showing that the reaction rate constant can have an appreciable effect on the back-mixing, and deriving an analytic expression for the particular case of a first-order reaction.

The horizontal box-type of mixer-settler is becoming increasingly popular in solvent extraction because of its high efficiency and versatility. A study of the effect of the mixer design on the efficiency of a pump-mix mixer settler was reported by Davis and Colven,<sup>63</sup> who found that higher efficiencies come from open impellers rather than from enclosed impellers at the same peripheral speeds. Nagata and Yamaguchi<sup>64</sup> made a very extensive study of extraction in mixer-settlers, using liquids with a wide range of density difference, reacting and non-reacting with each other. Their main conclusions were that the agitation mainly effects the drop size, and that the continuous-phase mass-transfer coefficients were little affected by operating conditions.

A multi-stage mixer-settler with reciprocating perforated plate agitators and inter-stage heat exchangers is now being marketed.<sup>65</sup> It is primarily intended for pilot-plant and small-scale production, and stage efficiencies very close to 100% are claimed.

## Viscous and non-Newtonian fluids

The power characteristics of different shapes and sizes of impellers for mixing non-Newtonian fluids were investigated by Metzner *et al.*<sup>66</sup> Their work is virtually a repetition on a large scale of the experiments of Calderbank and Moo-Young<sup>67</sup> and, since the results are almost identical, it would seem that the laboratory-scale experi-

ments of the latter give an excellent guide for the industrial design of mixers for non-Newtonian fluids. For pseudo-plastics and Bingham-type plastics, power requirements can now be predicted with almost the same accuracy as for Newtonian fluids.

Flow patterns in flour dough mixed in a laboratory machine have been studied with a carotenoid dye as a tracer<sup>68</sup> and the method should be applicable to larger machines. Flow patterns were also studied in a new type of mixer for viscous liquids as an aid to the optimum design of the critical dimensions.<sup>69</sup>

A range of mixers and masticators for rubbers, plastics and other viscous mixtures has been developed recently by Baker Perkins Ltd.,<sup>70</sup> who have just redesigned their small laboratory mixer. They also offer heavy-duty laboratory machines for the mastication of rubber, blending of plastics, etc. These mixers may be fitted with spring- or pneumatic-loaded rams to ensure operation at maximum efficiency.

The Bolling *Spiral-Flow* intensive mixer<sup>71</sup> has been especially designed to cope with the severe conditions encountered in the mixing of polyethylene and butyl rubber. By a system of floor lubrication, temperatures up to 400°F. can be catered for. All gears are removed from the mixer, permitting the use of interchangeable rotors. The mixer is available in five sizes from an 11-lb. laboratory mixer to a 600-lb.-capacity production model, the makers being Fawcett, Preston & Co. Ltd.

A new disperser for viscous solutions has been reported<sup>72</sup> and also improvements to standard designs.<sup>72, 73, 74</sup> A recent patent was granted for mixing a liquid additive with a continuous stream of viscous material.<sup>75</sup> A vacuum mixer for very stiff preparations has been described.<sup>76</sup>

For handling highly viscous materials the Moritz portable *Turbo-Reactor* is particularly suitable (see Fig. 4). This comprises a rotor with segments revolving within a specially designed stator, with segments which create an intense and controlled circulation.

For multi-phase dispersing and dissolving of plastics, inks, resins, paints, etc., the Cowles *Dissolver* has been developed. The effectiveness is due to the impeller. The narrow impeller vanes travel at speeds up to 6,000 ft./min. This direct mechanical action imparts extremely high velocity to material, creating a zone of intense turbulence immediately surrounding the impeller. The material

is forced rapidly outward through itself and, as the high-velocity material impinges on the surrounding slower-moving portions, hydraulic attrition takes place.

## Heat and mass transfer

In recent years there has been considerable investigation of heat transfer from jacketed vessels to agitated liquids contained in them. Ackley<sup>78</sup> has reviewed the published correlations for paddles, propellers, anchors and turbines as agitators. Unfortunately, there is as yet no single suitable equation for use with all types of agitators and vessel configurations. In another useful paper, Uhl and Voznick<sup>79</sup> have investigated the significant variables associated with anchor agitators which had not up till then been investigated, namely clearance, system geometry and width of blades.

Rate of solution of solids in an agitated vessel was the subject of a comprehensive paper by Barker and Treybal.<sup>80</sup> The motion inside the baffled tanks was turbulent and, as the authors pointed out, it is not surprising that the mass transfer is virtually independent of the Schmidt number.

Longitudinal mixing of fluids flowing in packed beds has been studied

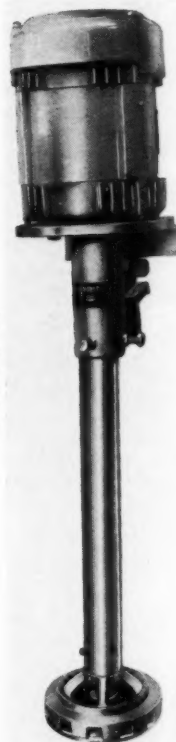


Fig. 4. Moritz portable 'Turbo-Reactor' particularly suitable for handling highly viscous materials. It can be fitted to any vessel in any position

by Cairns and Prausnitz,<sup>81</sup> Liles,<sup>82</sup> and Liles and Geankopolis.<sup>83</sup> The latter used a novel experimental technique to eliminate the end-effects in the bed and showed that the axial diffusion coefficient is independent of the length of the bed. Cairns and Prausnitz tested the model proposed by Einstein<sup>84</sup> and found it to be applicable. Binary gas mixtures were used by Robinson,<sup>85</sup> who found that for lengths of packing of less than 20 ft. the longitudinal diffusion coefficient is dependent on length, which seems to be at variance with the findings of Liles and Geankopolis.

Acrivos<sup>86</sup> proposes a simple method for estimating the effect of longitudinal diffusion and external mass-transfer resistance on the behaviour of a fixed-bed adsorption column. Axial mixing in a liquid fluidised bed was investigated by Cairns and Prausnitz.<sup>87</sup> The eddy diffusivity was strongly dependent on the density and concentration of the particles, and the results were consistent with the conception of a fluidised bed as being a transition between a packed bed and an open tube.

A numerical solution for the behaviour of an isothermal tubular reactor with various degrees of back-mixing was presented by Liang and Bailie,<sup>88</sup> while Hawthorn<sup>89</sup> has considered the effect of radial temperature variations on the axial mixing in a tube reactor.

### Miscellaneous

As is often the case, the unclassifiable material is quite interesting. While this article is mainly concerned with mixing, a recent paper on unmixing deserves notice. The usual mathematical description of the slow flow of viscous fluids within rigid boundaries indicates that this flow is reversible in time, and Heller<sup>90</sup> carried out experiments to prove this. Using solid tracers in a couette viscometer, he was able to bring back particles to their starting point by reversing the direction of flow after a given number of revolutions, although the pattern did smear in time due to thermal effects.

Briksmans<sup>91</sup> describes an electromagnetic method for stirring liquid metals and, although the metal with which he was particularly concerned was mercury, the method could well be applicable to other media.

Recent developments in mixing tubes for blending of liquids have been described above. Pawlowski<sup>92</sup> has shown that a mixing tube with injection apparatus suitably placed can

be made to divide into a number of cells so that the smooth tube is converted into a purely hydrodynamically formed multi-stage reactor.

Gutoff, who some years ago reported on the use of mixing tanks in continuous systems as a means of smoothing out cyclic fluctuations, has now considered the effect of agitator design on the efficiency of such an arrangement.<sup>93</sup> Unfortunately, his analysis is based on the so-called 'pumping rate' of the agitator used and, as this is not an easy quantity to measure, the utility of the method is at present doubtful.

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# Calcium Reactor Vessels for Argon Purification

By A. Knight,\* B.Met., F.I.M.

*After several years' service in argon purification plants, calcium reactor vessels fabricated in stainless steels were found to suffer from embrittlement of the inside surfaces which had been in contact with the calcium. This article describes experiments carried out to overcome this problem. It appeared, as a result, that completely satisfactory calcium reactor vessels could be fabricated using 18/8/Ti steel welded with 18/8/Nb steel; this could be lined with mild steel to obtain immunity from embrittlement by the heated calcium.*

A NUMBER of kinds of stainless heat-resisting steel were in use in the vessels containing calcium in the argon purification plants at A.W.R.E. In them, the calcium chips were maintained at a temperature of about 680°C. and at this temperature the oxygen and nitrogen impurities combined with the calcium and were removed from the argon. The vessels were heated externally by nichrome resistance elements to temperatures of about 700°C. Very occasionally the temperature of the vessels exceeded 750°C. because abnormal amounts of air were accidentally admitted into the system. In order to prevent the escape of gas which might contain radioactive and highly toxic particles, the pressure inside the calcium reactor vessels was maintained below atmospheric. Vessels which had operated for some years in contact with calcium had developed a hard and brittle surface layer. If cracks formed and opened, air would pass in and an intensely hot calcium fire could occur with the rapid destruction of the vessel and release of gas contaminated with radioactive and highly toxic particles. It was therefore necessary to carry out examinations of vessels which had been in service longest and to assess the suitability of the materials employed and, if necessary, carry out comparative trials of a selection of possible materials.

The vessels used in one plant, each about 1 ft. 3 in. in diam. by about 4 ft. high, were of elaborate construction and built up of many different steels: chromium-nickel steels and mild steel with bases which earlier had been of chromium-nickel steel were of *Nimonic* 75F, a nickel-chromium-iron 70/20/10 alloy. The body, main shell,

0.128 in. thick (10 s.w.g.), was of 25/20 chromium-nickel steel supplied by Firth Vickers as *Immaculate* 5. This steel of high alloy content, designed for use up to 1,100°C., was specified originally when it was thought that the working temperature in the shell might rise to 900°C., which was much higher than seven years' operating experience showed to be the case. The calcium container, fitted as a loose liner inside the main shell, was of mild steel. This mild-steel container was designed to facilitate the removal and disposal of spent calcium and was expendable.

The small vessels used by the A.E.A. at Aldermaston were about 1 ft. 6 in. high by 5½ in. diam. and had wall thicknesses in most cases of 0.064 in. (s.w.g. 16) with the rest 0.128 in. thick (s.w.g. 10). They were fabricated in stainless heat-resisting chromium-nickel 18/8/Ti steel, B.S. 1449/En 58B, with welding rods of 18/8/Nb steel, B.S. 1449/En 58F. The calcium was placed directly in the stainless-steel vessel.

## Attack on reactor walls

Occasionally, as the result of the accidental admission of air, the calcium partially burned and melted and during an exceptionally bad incident in a large reactor molten calcium flowed through the gas inlet holes in the bottom of the inner mild-steel container and made contact with the bottom plate of *Nimonic* 75F and with a thermocouple sheath of 25/20 chromium-nickel steel. The latter was destroyed and the former lost sharp edges by solution in the molten calcium. Although the mild steel was damaged, it had resisted attack more

effectively than the heat-resisting materials 25/20 steel and the *Nimonic* 75F.

Examination was carried out on small vessels which had been in use from 3,000 to 16,000 hr. with calcium in contact with the inside of the stainless steel. The microstructure of sections taken from various positions showed intergranular penetration to depths, varying with time of use, to

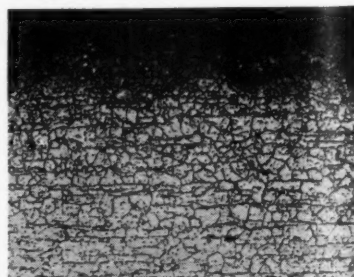


Fig. 1. Section of wall of calcium reactor vessel of 18/8/Ti steel after 16,000 hrs. use ( $\times 175$ )

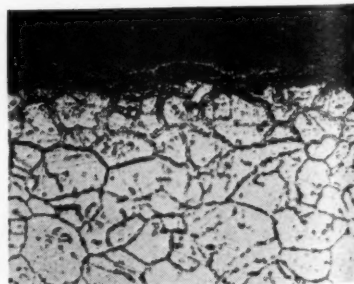


Fig. 2. Section of test strip of 18/8/Ti steel after 5,500 hrs. in calcium bed of reactor ( $\times 600$ )

\*U.K.A.E.A., Atomic Weapons Research Establishment, Aldermaston.

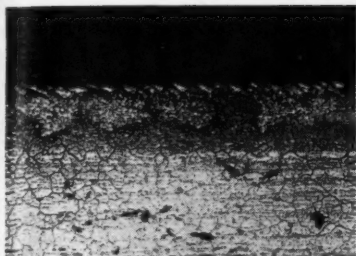


Fig. 3. 25/20 chromium-nickel steel. Thickness of surface layer affected 0.003 in. ( $\times 250$ )

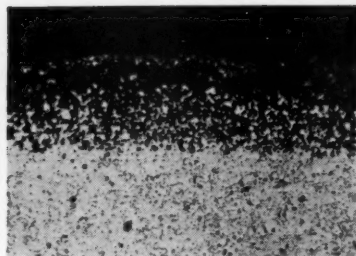


Fig. 4. 18/8/Nb chromium-nickel-niobium steel. Thickness of surface layer affected 0.004 in. ( $\times 750$ )

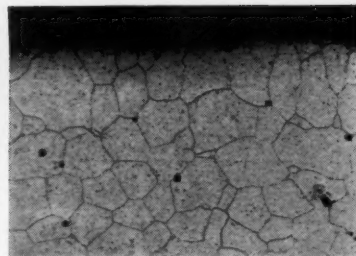


Fig. 5. 17% chromium steel. Resistant to attack ( $\times 100$ )

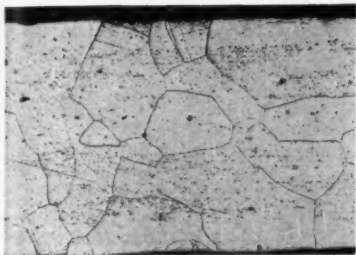


Fig. 6. Silicon-aluminium steel. Resistant to attack ( $\times 100$ )

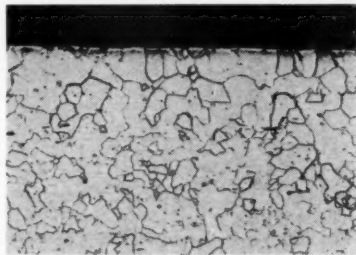


Fig. 7. Mild steel. Resistant to attack ( $\times 100$ )



Fig. 8. 25/20 chromium-nickel steel after 21,000 hrs. service ( $\times 250$ )

a maximum of 0.020 in. Typical microstructure of the affected layer are shown in Figs. 1 and 2. Although identification of the grain boundary precipitate was not attempted, it probably contained the intermetallic compound  $\text{CaNi}_5$ . Simple bend tests on strips cut from vessels withdrawn after long use showed the harmful effect of the calcium attack: bent inwards there was a high residual ductility shown in the outer surface layer, but bent outwards the ductility was shown to be considerably reduced and in the worst cases to less than one-sixth of the original high value. The actual values obtained by bending outwards over a radius of  $\frac{1}{16}$  in. were reduced in service in one vessel after 16,000 hr. from  $180^\circ$ , without breaking in the material as received, and in the same material taken from positions above the calcium level to bends of  $30^\circ$  or less to break.

In some designs of reactor vessels the solid calcium and, in case of accidental admission of an unusual amount of air, molten calcium also were in contact with the reactor vessel. In the design with an expendable liner, the reactor vessel at its hottest zone was exposed to attack by calcium vapour in argon.

#### Effect of solid and liquid calcium

In view of observations on vessels of various materials taken out of service and information on similar applications received from various sources, it appeared that nickel in any

material might be harmful. A series of materials was selected which, simultaneously exposed to calcium attack, would provide the evidence and indicate materials most suitable for fabrication of reactor vessels. Attack was slow at usual working temperatures of about  $680^\circ\text{C}$ ., but was speeded up by accidental admission of air, with consequent burning and partial melting of calcium. Therefore, accelerated comparative trials were carried out by immersing specimens in molten calcium at  $900^\circ\text{C}$ . for 8 hr. The apparatus was installed in a glove box containing circulating argon. Results are shown in Table 1 with the materials arranged in order of nickel content and, because welding is used in the fabrication of these reactor vessels, information on the suitability of each for this operation is included in column 3 of the Table. Typical microstructures are shown in Figs. 3 to 7, and the measured thickness of the affected surface layer is added to the table.

The harmful effect of nickel in this application is clearly shown. Nickel and the nickel-rich *Inconel* alloy dissolved completely in the calcium; the three nickel-containing steels (25/20, 18/8/Ti and 18/8/Nb) were adversely affected in microstructure and ductility. From the microstructures, it was seen that the surface layer affected was about 0.004 in. thick in all the steels containing nickel (Figs. 3 and 4). From the appreciable loss in weight, 16 mg./sq.cm. from the steel of highest

nickel, it was calculated that a further 0.001 in. was affected. As measured by the bend test, this steel, highest in nickel, suffered a 60% loss in ductility whereas the 18/8 steel lost 40% in ductility.

The nickel-free materials, including the four chromium stainless steels (Fig. 5 is of the 17% chromium steel), the silicon-aluminium steel (Fig. 6), the mild steel (Fig. 7) and the soft iron completely resisted attack; with these steels there was no penetration by calcium and no loss in ductility. This result with mild steel and soft iron is perhaps not surprising as all attempts to alloy calcium with iron failed.<sup>2</sup>

The heat-resisting silicon-aluminium steel which is non-scaling at sufficiently high temperatures to serve in these calcium reactors, completely resisted attack and should cost much less than chromium-nickel heat-resisting steels. There are, however, two objections to its use: first, its inferior mechanical properties at room and elevated temperatures as compared with the usual austenitic stainless steel and, second, that it has so far been made in small quantities only and any steel manufacturer would only supply if the quantity demanded was much greater than likely for this application. Possibly the lower ductility at room temperature and considerably lower strength at the temperature of working of the reactor vessels, about 6 tons/sq.in. against 18 tons/sq.in. for the heat-resisting chromium-nickel steels, could be easily compensated for by

Table 1. Effects of molten calcium at 900°C. for 8 hr. on various materials

Material	Nominal composition, %	Weldability	Thickness of surface layer affected, in.	Loss in weight, mg./sq.cm.	Ductility—180° bends to break†			
					Before exposure		After exposure	
					Longitudinal	Transverse	Longitudinal	Transverse
Nickel		Good	Completely dissolved	—	—	—	—	—
Inconel‡	80Ni, 14Cr, 6Fe	Good	Completely dissolved	—	—	—	—	—
Immaculate 5, stainless steel, A.I.S.I. 310	Chromium nickel 25/20 steel	Prone to hot crack	0.003	16	25	20	8	9
F.D.P.* B.S. 1449/En 58B	Chromium-nickel-titanium-steel, 18/8/0.5	Good	0.003	3.2	16	16	9	10
F.C.B.* B.S. 1449/En 58F	Chromium-nickel-niobium steel, 18/8/1	Good	0.004	2.1	—	—	—	—
F.I. 20* B.S. 1449/En 61	Chromium steel, 21 Cr	Poor welds; tend to be brittle	0	1.5	20	17	20	18
F.I. 17* B.S. 1449/En 60	Chromium steel, 17 Cr	Fair, but welds tend to be brittle	0	1.6	20	13	18	14
F.I.* B.S. 1449/En 56A	Chromium iron, 13 Cr	Fair; welds brittle	0	0.8	20	20	20	22
F.Al* A.I.S.I. 406	Chromium steel, 13 Cr, 4Al	Not recommended for welding	0	1.4	17	15	20	20
Fulmer Research Institute steel‡	2½ Si, 1Al	Good, but some loss of ductility	0	-0.1	6	3	10	4
Mild steel	0.07C	Good	0	-0.3	32	24	30	21
Soft iron	0.01C	Good	0	-0.6	31	20	23	21

\*Proprietary brand name of Firth Vickers Stainless Steel Ltd.

†The bend values were obtained in two directions at right-angles, respectively along and across the strip material

‡Nominal F, nominal composition 70Ni, 20Cr, 10Fe, as used for some base plates was not included in the laboratory trial, but the Inconel which was included was of similar composition

the use of increased wall thickness in the vessels because the internal pressures are only a few inches water gauge, but the supply position might prove more difficult.

In view of operating experience and the results shown in Table 1, it appeared that calcium reactor vessels fabricated from stabilised 18/8 steels and lined with mild steel would give satisfactory service.

#### Effect of calcium vapour in argon

On consideration of designs employing the above combination of materials it was pointed out that the argon gas leaving the calcium bed would be saturated with calcium vapour. Whilst it was shown that this did not adversely affect the chromium-nickel stainless steel above the calcium line where the steel and gas were relatively cool, it

might affect the vessel in the hottest zone where the working temperature was about 700°C. since the gas would flow back into the annular space between the stainless steel and its mild-steel liner. It was decided, therefore:

- (1) To examine the microstructure of a 25/20 stainless-steel vessel which was in use in one plant for 21,000 hr. under the conditions proposed in the new designs, i.e. with a mild-steel liner sealed to the outer vessel at its lower argon inlet end only.
- (2) To expose chromium-nickel steels and mild steel to argon saturated with calcium vapour at 900°C.

The microstructures of the inner surface of the 25/20 steel vessel after long service showed no measurable

attack (Fig. 8). This photograph, taken on a section from the hottest zone, showed slight corrosion caused probably by exposure of the vessel to the weather for about a year after removal from service.

Laboratory trials in an argon-filled box were carried out by suspending steel of the same composition, the 25/20 steel, and also the 18/8/Ti, mild steel and soft iron, just above the molten calcium held in a mild steel covered pot at 900°C. in one trial for 8 hr. and in another for 240 hr. Under these conditions the partial pressure of calcium vapour would be about 0.1 mm. mercury, much higher than at the stainless-steel surface in the argon purification plants using reactor vessels fitted with mild-steel liners. After the 240-hr. test, the specimen of 25/20 steel was coated with calcium and it was probable that a splash of calcium under the clean conditions had spread over the surface. Micro-examination showed even penetration to a depth of 0.003 in. There had been no measurable penetration in this 25/20 steel after 8 hr. (Fig. 9). There was, at the conclusion of the trials, no penetration into 18/8/Ti steel (Fig. 10), mild steel or soft iron.

As a result of these experiments, the following conclusions were made:

- (1) The effects of calcium on the heat-resisting chromium-nickel 25 20

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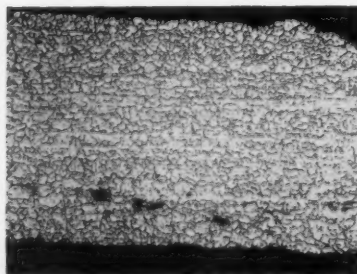


Fig. 9. 25/20 chromium-nickel steel exposed to argon saturated with calcium vapour at 900°C. for 8 hrs. (× 60)

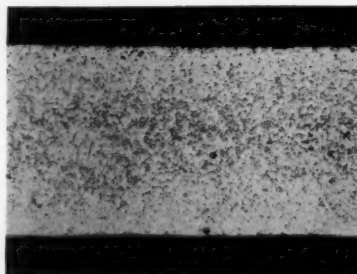


Fig. 10. 18/8/Ti exposed to argon saturated with calcium vapour at 900°C. for 240 hrs. (× 60)



# Applications of Computers to Process Control

## Part 2

By J. F. Roth,\* B.Sc., A.M.I.E.E.

*Having described the structure of a data processing system in Part 1, the author, in this second article, shows how such a system may be combined with a digital computer to form a computer-controlled installation. Methods of utilising these installations for process control are given and it is shown that, despite their high initial capital cost, their use should entail considerable saving in processing costs and improvement in efficiency.*

**B**Y combining the basic logger concept with a digital computer, an extremely versatile and powerful machine can be provided which is more than adequate for the present and even future demands of industry.

However, there is always a shadow on a silver cloud, and on this one it is a matter of cost. Yet even this is not really true for, although there is an obvious step function in the cost between a fixed programme system and a computer-controlled installation, due to the addition of the computer, the capacity of the latter system for improving the process it is controlling will far outweigh that increase which will eventually be retrieved in plant savings. As an example, a computer-controlled system recently installed in a steel slab mill to reduce wastage will recoup the capital cost of the equipment within a year if the throughput is increased by about  $\frac{1}{2}\%$ . Or in an ammonia-producing plant an improvement of 1% in the yield may show a saving of £20,000 per year.

When considering an installation of this kind it must be remembered that one is not simply mechanising the process of data acquiring, but one is at the same time providing for the full automation of the plant. This will provide, amongst many advantages, for improved efficiency and thus lower operating costs.

With modern plants the size they are, the cost of full automation runs into figures which can be quite frightening, but then the savings which can be made are similarly amazing. Fig. 6 shows the computer-controlled data processing system, and Fig. 7 is a view of the author at the control desk of a data processing system. The left-hand panel contains the alarm display, and the associated

alarm printer is adjacent to it. The centre panel houses the clock and demand visual displays. On the right-hand panel are the manual command switches, again with the associated printer next to it. From this console full supervisory control of the plant can be attained.

### Organisation of computer system

The power and flexibility of a computer-controlled system is derived purely from the use of a digital computer. This provides a system centre which is capable of performing logical operations to control the input selection, arithmetic and sorting operations on the data measured and the generation of suitable control signals, all of which are performed at an extremely high speed. However, the internal organisation of the computer must be such that it is capable of operation on a peak load (e.g. when many things are happening on a plant at the same time, for example under alarm conditions). This may sound extremely obvious, but it does represent a serious limitation which on a very large plant may be quite embarrassing. The corollary of this requirement is that, when little is happening on the plant (for example, no alarms and only normal scanning going on), the computer is idle for most of the time.

There is, however, a way through this apparent *impasse*. If one examines the operations which have to be performed by the computer system it will be found that they can be divided into various levels of priority. There are those—for example, alarm indications—which obviously have a high priority, and others, like changing the period between automatic log print-outs, which have a very low priority. Hence a system of time sharing can be used

which spreads the load and ensures that the computer will never be overloaded. The method used to accomplish this is basically to provide for the interruption of one task if another of higher priority occurs whilst the lower priority one is being dealt with.<sup>1</sup>

One practical solution is to use a 'control word'. The various tasks performed by the system are each allocated a unique position in the control word register, and can thus indicate their condition to the computer. The programme has to be so designed that it repeatedly examines the control word to see what task has to be performed next, or to see whether a particular facility is available for use.

The following examples will illustrate the use of the control word. The typewriter for printing the log has a 'busy' contact on its mechanism to signal when it is actually printing a character. This is used to place a marker in the respective position in the control word. Now, after having prepared a character for output, the control word is examined at the requisite position to see if it is possible to print a further character. If it is, the character is printed. If it is not, the character is held in the store, and the programme proceeds to do the next operation, after which it will return to the print-out routine, test the control word again and proceed as before. Thus the computer is prevented from being held up and is able to perform an alternative task.

Another example would be the manual commands. These are requests by the operator for the system to perform certain functions which may be interrogatory or alteration of

\*Panellit Ltd., Elliott Automation Group.

operating procedure, *e.g.*

- (a) Change the time interval between successive routine logs to that set on decimal switches.
- (b) Change the alarm level to the value set on decimal switches.
- (c) Print out current information regarding the input point set on decimal switches.

One set of switches are provided for selecting the required command, and another set or more for associated data (*e.g.* point number or time interval). To initiate the command, a push button is provided which is operated when all the relevant switches have been set. This action places a digit in the control word. Periodically, the programme looks at this digit to see if a manual command is waiting to be obeyed. If so, it examines the command switch to see if the priority of the command is such that it should be obeyed.

At first sight it would appear that a lot of time is wasted by repeated examination of the control word digits. However, it should be noted that it is only necessary to repeatedly examine at regular intervals those digits whose presence or otherwise is completely out of control of the computer—for example, the manual command push button. On all other occasions the programme knows when it is necessary to perform the examination, for example it is only necessary to see if the typewriter is available to print a character when a character is ready to be printed.

### Priority interrupt

An alternative approach to solving the time-sharing problem would be to use an 'interrupt' arrangement.<sup>5</sup> The control word approach uses a 'busy' system, whereas the interrupt is the

inverse of this, *i.e.* a 'ready' system. In the busy system the device concerned indicates it is busy when it is required for use, but the ready system asks for attention as soon as it becomes available. Thus referring to the typewriter example mentioned above, it can be arranged that, after presenting one character for printing, the next character is not even prepared until the typewriter signals that it is ready for it. The system employed is that, when the device signals that it is available, the programme is immediately interrupted, providing that the priority associated with the interrupting device is higher than that associated with the current programme. The first operation of the programme when the interrupt occurs is to store the point in the programme where the interruption occurred. The interrupt is then dealt with and, when complete, the programme reverts to the point in the original programme where it was interrupted. One can, of course, have interrupts upon interrupts, all of which the programme must take care of. Thus, there is quite a large amount of programming organisation in order to ensure correct transfers from a programme and back again. In addition, sub-routines and working space cannot be shared between different operations, as a sub-routine can be interrupted at any point. The most rewarding device for application of the priority interrupt facility is where signals or operations occur at random. For example, the manual command request push button which the operator can press at any time, or a timing pulse for synchronising the operation of the system to real time.

There is quite evidently a place for both control word and priority inter-

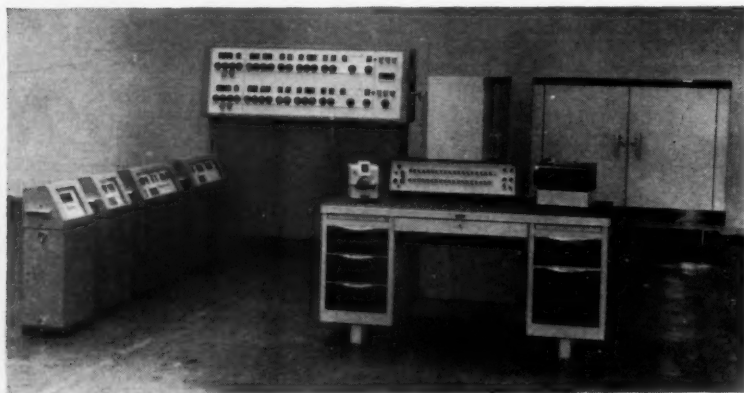
rupt time-sharing methods in any complete system, and normally they would be used in conjunction with each other.

### Alternative programme

The basic organisation behind time-sharing is that, when nothing of higher priority is required to be done, the programme operates on the lowest priority, which could well be going round a loop looking for something to do. This means that the computer is in effect doing no useful work. Hence, if there is sufficient storage space, another programme can be run on the computer during this wasted time. The proviso is that this programme is, and always is, of lower priority than the main programme. Thus the organisation of this second programme must be such that it is broken up into small sections. Between each section reference is made to the main programme to determine if this requires attention. Alternately, this organisation can be achieved by using priority interrupt techniques.

Theoretically, this second programme could have no relation whatsoever to the main programme. It could be stock control or a wages programme, but this combination would not be advised. Even though the second programme had been fully tested, there is still a remote chance, perhaps as a result of operator error, of the two programmes interfering with each other and the computer stopping. No plant manager would risk another department causing such a breakdown. However, it does show what can be done, for the secondary programme could well be an analysis programme of the information collected by the system from the plant, and in this case would be fully under the control of the plant manager and would be giving him additional operating information.

Using this facility, experimental on-line investigation of plant operating conditions can be undertaken without interfering with the main programme. From the results and information produced by this programme, it may well be found, for example, that some relationship seems to apply to a few of the measured or calculated values. An alternative programme can then be prepared to check this. From the control console the operator can, using the manual commands, read in the programme in parallel with, and without affecting the operation of, the main programme. Again using the manual commands, he can start the alternative programme (and similarly stop it).



[Courtesy: Elliott Bros. (London) Ltd.]

Fig. 6. Computer-controlled data processing system. On the left are the consoles placed in the plant for inputting information and displaying control information. The computer is housed in the two cabinets at the rear, and can be controlled from the desk in front

No doubt the first attempt will not give the required results, and another alternative programme will have to be prepared. This can be entered again and eventually, using a trial and error technique, correct operation will be achieved. At this stage the alternative programme can be incorporated in the main programme.

As can be appreciated, this approach allows gradual examination and analysis of plant to be undertaken so that a complete control system can be evolved in successive stages.<sup>6</sup> When the aim is closed-loop operation, it is possible to check each sub-loop in this way and ensure that the correct control information is being produced before each sub-loop is actually closed.

### Plant control

Where a computer-controlled system is to be used for plant control, two methods of operation are available, both of which provide indirect operation. Firstly, instructions can be presented to the plant operator for him to adjust the specified controls. Secondly, the system can directly adjust the set points of conventional controllers. It is, of course, possible to employ direct control of plant equipment, but this might not provide for safe operation of the plant in the event of a failure in the process control equipment. In the indirect method, safe operation in the event of failure is provided for as, although it is almost certain that optimum operating conditions will not be maintained, all controls will remain where last set. But at least the operating staff know that the plant is safe, and can rapidly change over to manual operation.

### Safety

It is very evident that an extremely high order of reliability is essential for a computer-controlled system.<sup>7</sup> One advantage of digital systems is that by their very nature they are either working or not and there is rarely an intermediate condition when the equipment appears to be working but is giving incorrect information. To cover this type of failure, a series of checks can be incorporated in the programme to give immediate fail safe type of indication in the event of any malfunction.

An alternative approach is to duplicate sections of the equipment, although this requires a method of detecting an error in one piece of the equipment and then switching over to the standby. To ensure that the standby is operational, the two sections could be used alternatively under nor-



(Courtesy: Elliott Bros. (London) Ltd.)

Fig. 7. The author at the control desk of a typical computer-controlled data processing system

mal conditions, but as soon as a failure in one is detected, the other would be used exclusively. This arrangement requires more equipment and does not protect for failure of the change-over system. Another difficulty is in deciding which sections to duplicate, as there is often little reason to suspect one part as being less reliable or less likely to render the system useless than any other. Taking this argument to its logical conclusion, three equipments are required using a 'two out of three' principle.

Failure of the mains supply is, of course, a hazard which will switch the control system off. Often it is at just such a time as this that the control system should be working. This facility can be provided by running the system from batteries which are being trickle-charged.

With solid-state systems this arrangement is most attractive as power supplies become much simpler. Thus, when the mains fails, the control system carries on from the battery. This period allows plenty of time for alternative supplies to be provided.

### Optimisation

One of the biggest drawbacks to the installation of data processing systems to complex operations like oil distillation, is that the operating laws for the process must be known so that a programme can be written. A simple method of applying automatic control to such an unknown process can be achieved by using an optimiser.<sup>8</sup> With this system, one of the output parameters is taken as a reference and facilities are made for the optimiser to be able to vary the process variables one by one by small amounts. The

procedure is that one of the inputs is varied and, after a suitable time interval, selected as a result of knowledge of the time constant of the process, the output reference is examined. If this has improved, then the process is repeated until a peak is reached. Then the same procedure is repeated with the next variable. Thus, by successive approximations, the peak is attained which by normal standards would be an unstable point. The process can be compared with a blind man trying to reach the top of a hill by first of all walking in one direction until he starts to descend and then changing his direction and walking again until he starts to descend. By constant repetition he will get to the top of the hill. It should be noted, however, that the blind man may only find the top of a hill on the way up and the same thing applies to the optimiser.

Thus, it must be started near the peak, and limitations placed on the variables so that they cannot be shifted too far from the optimum.

Although this approach looks extremely attractive, there are many practical problems which have to be solved before a successful installation can be produced. These revolve mainly around the difficulty in selecting the amplitude of the variations to be applied to the process variables, and in the time delay between making a change and sampling the reference parameter.

### Conclusion

The great improvement in methods of monitoring and control of industrial plants which are required in order to further improve their efficiency and



productivity can be provided by applying computers to the problems of process control. At present the development of the equipment is far more advanced than the knowledge of how to apply it to a plant, and the balance will only be rectified as more systems are installed and experience gained on how to use them to the best advantage. The first stage is to install data acquirers and, if sophisticated ones are chosen, they can not only determine how the plant operates, but can be adapted later to control it. The next stage is to use all the accumulated information and to design, using a computer, the next generation of the plant itself. It appears that the chemical industry offers many examples for this style of approach for it would seem that, in this way, for example, storage vessels that account for such a large proportion of these plants could be dispensed with.

In large plants, a combination of fixed or semi-fixed programme routines being used in conjunction with a central computer-controlled system can offer many advantages, especially with regard to cost. This echelon formation

provides for maximum usage of the central computer, using time-sharing techniques to supervise the satellite machines, which would consist of fixed programme systems carrying out routine operations. In fact, using this technique, a managerial pattern can be evolved.

To achieve satisfactory operation of a computer-controlled data processing system when applied to a practical process control problem, requires full co-operation between plant personnel and the equipment manufacturer.<sup>6</sup> The amount of joint study which is required to produce the system specification represents a considerable portion of the total effort involved in engineering and commissioning the system and, consequently, the time required can be quite large. It often happens that, as a result of this detailed investigation, methods of improving the existing instrumentation and control methods come to light, and recognition of this fact should be borne in mind when the initial decision to embark on a computer system is under discussion.

Future progress in this field will be

extremely rapid, and the problem of keeping up to date will be immense. In fact, one is reminded all too forcibly of the Queen's comment to Alice in 'Alice Through The Looking Glass', 'Now here, you see, it takes all the running you can do to keep in the same place. If you want to get somewhere you must run at least twice as fast as that'.

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## Calcium Reactor Vessels for Argon Purification

(Concluded from page 496)

and 18/8 stainless steels, at about 700°C., in argon purification plants were intergranular penetration and embrittlement.

(2) Accelerated laboratory tests in which many materials were immersed in molten calcium showed that all nickel-bearing alloys were adversely affected in structure and properties in proportion to their nickel content. On the other hand, nickel-free materials, including chromium stainless steel, a silicon-aluminium steel and mild steel, were completely resistant.

(3) Argon saturated with calcium vapour at 900°C. made no significant effect on the chromium-nickel steel, including the 25/20 steel which was the steel most susceptible to attack by molten calcium.

(4) The chromium-nickel 18/8 steel stabilised by titanium in the plate and by niobium in the welding rod was satisfactory for fabrication of the outer vessel of the calcium containers of the argon purification plants. According to Aldermaston experience and the steel manufacturers' recommendation, it was suitable for continuous use in air at 800°C., i.e. about 100°C. above the normal working temperature of the plants.

(5) The weldability of the highly alloyed wholly austenitic 25/20 chromium-nickel steel presented a problem in hot cracking in 1952, when the earliest Aldermaston plant was erected. Also, none of the plain chromium steels are capable of giving the necessary high-quality crack-free ductile welds. The cheap silicon-aluminium heat-resisting steel, although giving acceptable welds, was not readily available. On the other hand, the stabilised 18/8 chromium-nickel steel has excellent weldability having the right amount of ferrite in the austenite matrix to prevent the formation of cracks in and about the weld. There would be no difficulty in producing the quality of welds required in the liner of mild steel or soft iron.

(6) A mild steel would provide a cheap liner material satisfactory in its fabricating quality and complete resistance to calcium attack.

(7) The safe working life of a calcium reactor assembly made of chromium-nickel 18/8/Ti steel, B.S. 1449/En 58B plate of 10 s.w.g. (0.128 in.) welded with rods of 18/8/Nb steel, B.S. 1449/En 58F with a mild-steel liner, should be at least 50,000 hr.

#### Acknowledgments

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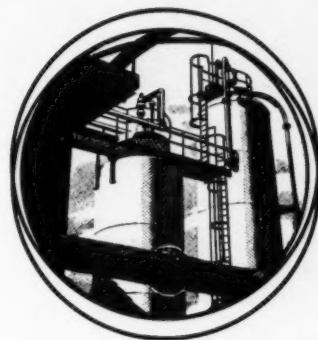
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# Materials of Construction for Chemical Plant

## RUBBER

By I. L. Hepner, Ph.D., A.C.G.I., A.M.I.Chem.E., A.I.R.I., F.N.C.R.T.



*The nineteenth article in the series 'Materials of Construction for Chemical Plant' is on rubber. Previous articles in this series have included PVC, lead, nickel, stainless steels, graphite, polyolefines, copper, timber, platinum, titanium, aluminium, reinforced plastics, cast iron, mild steel, silver, glass, ceramics and new metals. Rubbers and ebonites are probably the oldest polymeric materials used for chemical plant construction. Their main present-day uses are restricted to lining of tanks and other plant equipment, thus providing excellent resistance to highly corrosive chemicals. For this purpose, rubbers are considerably cheaper than the range of high-alloy steels.*

WHEN assessing the suitability of rubbers as materials of construction for chemical plant, it is important to consider the term rubber as comprising a family of long-chain polymers which all have the capacity to undergo large deformations at atmospheric temperature without rupture. This property, usually described as rubber elasticity, is not due to a particular chemical constitution of the rubber molecule but rather to the arrangement of the polymeric chain into a highly randomised coiled network which is cross-linked at specific points. Rubber elasticity is exhibited not only by natural rubber of the *Hevea brasiliensis* type (1 : 4 cis-polyisoprene), but also by synthetic rubbers such as styrene-butadiene, neoprene and styrene-acrylonitrile. Ebonites, on the other hand, which are rubbers with a large proportion of intramolecular-bound sulphur, are not elastic at all; they rather resemble the brittle plastics like phenol-formaldehyde or polystyrene.

Rubbers and ebonites can claim to be the oldest polymeric materials used in the chemical plant industry. Their applications in this sphere have mostly

been confined to linings bonded on to metallic plant which improve the chemical resistance and decrease the rate of corrosion of the plant unit. The idea of bonding a layer of rubber to a metal surface, in fact, dates back to 1855 when J. H. Johnson was awarded a patent for making spinning components out of metal covered with hard 'india-rubber'. Johnson claimed that this combined the advantages of the strength and durability of the metal with the 'non-liability to oxidation' of 'india-rubber'.

This article will attempt to trace the historical origins of natural and synthetic rubbers and ebonites as materials of construction for chemical plant and to discuss the chemical, physical and mechanical properties of rubbers and their particular suitability for fabricating and lining chemical plant.

The main source of natural rubber is the latex flowing through the vascular system of the *Hevea brasiliensis* tree (originally a native of the Amazon valley). There are many other species of plants that can produce good-quality rubber; several of them are to be found in Liberia, the Congo, India and countries in South-East

Asia. It was only with the introduction of the *Hevea* species into Malaya by the British government, that a large-scale attempt was made to systematically cultivate a uniform type of rubber in plantations. As a result the cultivated rubber plant was im-



Fig. 1. A vat for processing uranium, titanium, vanadium and cobalt, lined with neoprene

proved considerably so that nowadays the world production of plantation rubber is almost 2 million tons p.a. The latex rubber in the *Hevea* tree is an emulsion consisting of cis 1:4-polyisoprene globules dispersed in a liquid serum of water, proteins, lipids, quebrachitol and inorganic salts. Although some types of rubber articles can be fabricated directly from the latex (for example, rubber foam and dipped rubber gloves, etc.), by far the largest proportion of rubber used in industry is first converted to the dry state, by coagulating the rubber particles in the latex with acid or heat. The rubber particles in the latex are thus precipitated and then rigorously dried and in some cases smoked. The resultant rubber is in the form of either pale *crêpe* or 'smoked sheets', which are subsequently shipped from the Far East plantations to rubber manufacturers all over the world.

### Historical

Ever since Williams in 1860 discovered that the building brick of the rubber molecule consists of isoprene,  $C_5H_8$ , chemists have considered the possibility of obtaining rubber synthetically. Tilden in 1910 reported the spontaneous polymerisation of isoprene, but it was not possible to reproduce this on a large scale until the advent of stereo-specific catalysts in 1957. It was found by these early workers that other polymeric materials with rubber-like properties could be synthesised much more readily.

As a result of the first world war, the first large-scale production of synthetic rubber occurred. In Germany at that time there was a desperate shortage of rubber and it became vital to produce it at any cost. The Bayer company eventually succeeded in synthesising dimethyl butadiene by the pinacone process; this was converted into methyl-rubber (which had already been discovered by the Russian Kondakov in 1901).

Only a small quantity of these rubbers were produced because they were of poor quality, largely due to the fact that at that time the reinforcing effects of carbon black were unknown. For this reason, production of this type of rubber was discontinued as soon as the war ended. During the middle 1920s it was realised that it was more important to make rubbers having properties which natural rubber does not possess, such as oil and solvent resistance. As a result, work was continued in Germany on sodium polymerisation and this led eventually to the discovery of *Buna S* and *Buna N* by

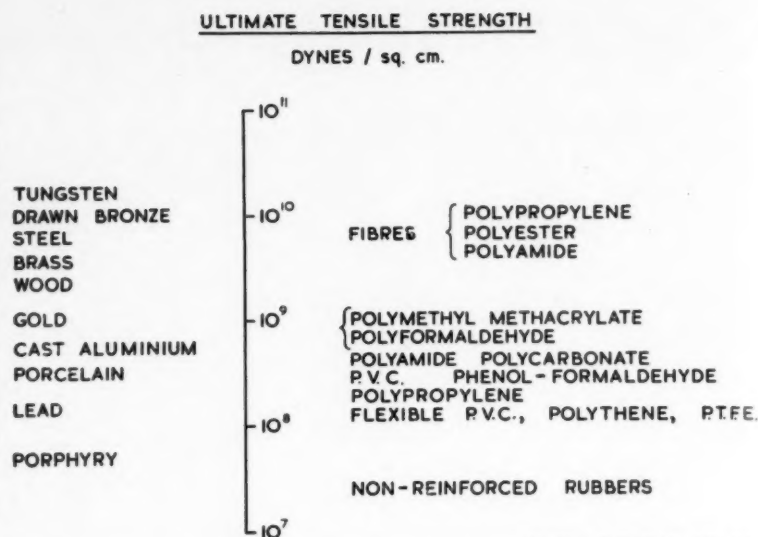


Fig. 3. Ultimate tensile strength of rubbers compared with plastics fibres and metals

the emulsion polymerisation technique, which made possible co-polymerisation of two or more monomers.

Polychloroprene (neoprene) was developed by the Du Pont Co. in 1925, when a young Du Pont chemist, Dr. E. K. Bolton, heard a paper presented by Father J. A. Nieuwland, professor of organic chemistry at the University of Notre Dame, before the American Chemical Society. Father Nieuwland was not even casually interested in the quest for a satisfactory synthetic rubber; instead, he was reporting on his research on acetylene gas. Dr. Bolton was deep in the subject of synthetic rubber.

After the meeting, Father Nieuwland suggested to Dr. Bolton that he visit the Notre Dame laboratory to obtain more information about the university's research on acetylene. Back in their laboratory, Du Pont chemists found that Father Nieuwland's process to make divinylacetylene could be modified to produce monovinylacetylene, made by the union of two molecules of acetylene gas. They

found that treatment of monovinylacetylene with hydrochloric acid resulted in a previously unknown chemical which was called chloroprene. It was soon found that chloroprene could be converted into a rubber-like solid superior to natural rubber in many respects. This product became known by the generic name neoprene.

Simultaneously other groups of oil-resisting products were developed in the U.S.A. by Patrick (*Thiokols*) and in Switzerland by Baer (*Perbunans*).

### Second war crisis

At the outbreak of the second war, it was realised that a rubber shortage would soon develop and in the U.S.A. and those companies with a knowledge of synthetic rubber co-operated on the production of several general-purpose and special-purpose rubbers. The rubbers then produced were GR-S, GR-M (neoprene), GR-I (butyl rubber) and GR-A (*Buna N*). The largest output was devoted to GR-S, the styrene-butadiene rubber that could satisfactorily replace natural rubber in tyres and general mechanical goods. Nowadays, 16 years after the end of the war, the quality of synthetic rubbers has been improved to such an extent that they can replace natural rubber for many applications and, in addition, can be used for applications that are unsuitable for natural rubber such as, for example, oil seals, high-temperature components, etc.

### Emulsion polymerisation

Most synthetic rubbers are generally obtained by the liquid-phase emulsion

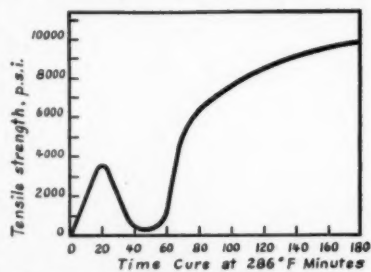


Fig. 2. Increase of tensile strength with time of cure for natural rubber ebonite



polymerisation technique. This technique has two advantages, namely that the viscosity of the emulsion hardly changes throughout the reaction, hence thermal control is simple and, as a result, monomers with different speeds of polymerisation can be effectively co-polymerised. The polymerisation is carried out by emulsifying the monomer or mixed monomers with a suitable emulsifier to which is added a chain modifier, an initiator, sequestering agent, electrolyte, short-stop agent and antioxidant. Polymerisation is terminated as soon as the required polymer chain length has been obtained (the degree of polymerisation determines the physical properties of the rubber). The rubber, now in the form of a latex, is coagulated in a similar way as natural rubber latex so as to obtain rubber in the dry state.

Synthetic rubbers obtained by emulsion polymerisation include SB-R (styrene-butadiene rubber), nitrile rubber (butadiene-acrylonitrile) and neoprene (polychloroprene).

### Bulk polymerisation

Bulk or ionic polymerisation is used to a lesser extent in the production of synthetic rubbers. Although this technique was first developed in Germany (as mentioned previously), interest in it was renewed when it was found that the structure of the rubber could be influenced by the addition of various metallic catalysts. The culmination of this work has been the very recent production of natural synthetic rubber, 1:4 cis-polyisoprene by bulk polymerisation, using a mixture of metals which caused the polymerisation to proceed in an ordered stereo-specific manner.

Another rubber obtained by bulk polymerisation is butyl rubber (polyisobutylene-isoprene), which is produced by the instantaneous co-

polymerisation at low temperatures ( $-150^{\circ}\text{F.}$ ) of a mixture of 96% isobutylene and 4% isoprene. The catalyst is a dilute solution of aluminium chloride in methyl chloride, cooled with liquid ethylene. The polymer as a slurry is dropped into the water to flash off diluent and any unchanged monomers. This rubber forms no latex, but is initially produced in the dry form.

### Compounding

Pure rubber in its natural state is very tough and elastic. It has a tensile strength of about 2,000 to 3,000 p.s.i. and an elongation of about 900%. By suitably compounding with a variety of compounding ingredients or fillers, a greater tensile strength may be obtained as well as a reduced elongation at break. For example, a composition suitable for lining tanks may contain only 60 to 80% rubber and will elongate not more than 500 to 600%; its tensile strength will be considerably higher than 3,000 p.s.i. Thus, by reducing the rubber content and increasing the proportion of reinforcing filler such as carbon black or china clay, the tensile strength of the rubber is increased and the elongation is reduced until eventually the rubber hardly stretches at all and resembles leather. Besides these reinforcing fillers, other compounding ingredients such as chalk, mineral oil and factice, may be added to rubber in order to reduce its high viscosity and enable it to be formed in extruders or calenders into the required shape of the article. Antioxidants are also added to improve the ageing and weathering resistance of the rubber. Finally, sulphur and organic accelerators (for example mercaptobenzthiazole) are added to the rubber compound in order to promote vulcanisation. Vulcanisation is a chemical reaction of the sulphur with the rubber

polymer chains, whereby the sulphur forms cross-links between adjacent polymer chains to give a three-dimensional elastic network. The shaped rubber article is normally vulcanised or cured by heating with steam, either in an autoclave or in the open atmosphere.

### Ebonite

For 'soft' rubber or elastic rubber, discussed above, the process of vulcanisation has proceeded only to a small extent—the amount of sulphur combined with the rubber usually varies from 1 to 3%. However, a molecule of rubber can theoretically combine with up to 47% sulphur. If about 40 parts of sulphur are added to 100 parts rubber and the mixture is subjected to prolonged heating, a hard brittle product, ebonite, is formed.

Fig. 2<sup>1</sup> shows the increase of tensile strength with time of cure for a typical natural rubber ebonite. It will be seen that, after a short time of cure, a typical soft rubber vulcanisate is obtained, but for more prolonged cures the tensile strength increases significantly. Ebonites are much harder, more brittle and considerably less elastic than soft rubbers. They are normally produced in the basic forms of sheet, rod, tube and extruded sections from which the finished form can be completed by machining. Ebonites can be made from most synthetic rubbers except butyl rubber, neoprene and chlorosulphonated polyethylene (*Hypalon*).

Prior to the large-scale use of plastics for chemical plant units, ebonites were used extensively for fabricating chemical plant equipment such as pump casings, propeller shafts and blades and other units which are in contact with highly corrosive chemicals.

### Physical properties

Normal engineering materials other than rubber are usually used up to their limit of proportionality. Up to this point Hooke's law is obeyed, i.e. the strain is proportional to the applied stress. This is referred to as normal elasticity, such as is, for example, exhibited by steels. Rubber-like materials are characterised by a different form of elasticity known as rubber elasticity, which is much larger deformation without rupture, and the stress required to produce this deformation is of a much lower order. Unlike the materials exhibiting ordinary elasticity, the stress-strain relationship is linear for only a small portion of the total deformation. The initial modulus of elasticity is of the order of 10 kg./sq.cm. In Fig. 3 the

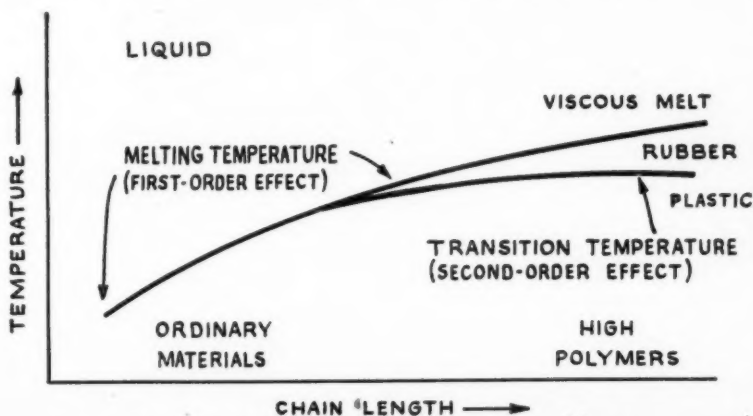


Fig. 4. Schematic chain length versus temperature diagram for linear polymers

ultimate tensile strength of a variety of materials—rubbers, plastics, ferrous and non-ferrous metals and fibres—are compared. It can be seen that rubbers have a much lower ultimate strength than plastics, fibres and metals.

An examination of elastic properties of a typical range of high polymers over a wide temperature range shows that all polymeric materials exhibit rubber-like elasticity in the temperature range above the second-order transition point. Fig. 4 shows a graph obtained by plotting chain length against temperature. From this curve it can be seen that, at a certain minimum chain length, polymers which are glass-like at room temperature will pass through a second-order transition at some higher temperature and subsequently exhibit rubber-like properties. Therefore the description of a material as a rubber or a plastic depends on whether its second-order transition occurs above or below normal temperatures. Thus natural rubber has a transition point of  $-10^{\circ}\text{C}$ . Below this temperature, it is tough and

brittle. Polystyrene, on the other hand, is tough and brittle at room temperature, but its transition point is  $70^{\circ}\text{C}$ .; above this temperature it becomes rubbery and soft. In the case of PVC, the high transition point from the glassy to the rubber state (which also occurs at about  $70^{\circ}\text{C}$ .) can be reduced to atmospheric temperature by the incorporation of plasticisers. Hence plasticised 'soft' PVC is really the 'rubber' state.

Table 1 gives the physical, mechanical and chemical properties of nine commonly used rubbers. By examining the tensile strength and hardness range of each rubber, it can be seen immediately whether this rubber is suitable for the duty required. The vulcanising properties give an indication of the ease or difficulty in curing an article. Thus, butyl rubber is more difficult to cure than SB-R. Ease of curing is of particular importance for rubber linings. Thus, with natural rubber or SB-R, the lining will cure quite rapidly, but some trouble may occur with butyl rubber,

and considerable care must be devoted to compounding the correct vulcanising ingredients to the butyl stock so that a reasonably rapid and permanent cure is obtained.

### Chemical resistance

Natural rubber has a wide range of chemical resistance including most acids, alkalis and salts used in industry. However, it cannot be used satisfactorily in contact with mineral oils and solvents or with strong oxidising acids such as nitric, chromic and concentrated sulphuric acid. It is for this reason that synthetic rubber has found such wide applicability in industry. Neoprene, for example, can resist products that would rapidly attack natural rubber such as caustic soda, fluorides, ammonium nitrate, greases, oils, fatty acids and other aliphatic hydrocarbons. In addition, neoprene can also withstand processing temperatures up to  $250^{\circ}\text{F}$ .

For even greater resistance to oils and aromatic hydrocarbons, nitrile rubber is recommended; however, its

Table 1

Properties	Natural rubber (polyisoprene)	SBR (butadiene styrene)	Butyl (isobutylene isoprene)	Thiokol (organic poly-sulphide)	Nitrile (butadiene acrylonitrile)	Silicone, poly-siloxane polymer	Neoprene (poly-chloroprene)	Viton (co-polymer of vinylidene fluoride and hexafluoropropylene)	Hypalon (chloro-sulphonated polyethylene)
TENSILE STRENGTH (p.s.i.):									
Pure gum .. .. .	>3,000	<1,000	>1,500	—	<1,000	<1,500	>3,000	>2,000	>2,500
Black loaded stocks ..	>3,000	>2,000	>2,000	—	>2,000	—	>3,000	>2,000	>3,000
HARDNESS RANGE (Shore Durom. A)	30 to 90	40 to 90	40 to 75	35 to 80	40 to 95	40 to 85	40 to 95	60 to 95	40 to 95
SPECIFIC GRAVITY (base material) ..	0.93	0.94	0.92	1.34	1.00	—	1.23	1.85	1.12/1.28
VULCANISING PROPERTIES	E	E	G	F	E	—	E	G	E
ADHESION TO METALS .. .. .	E	E	G	P	E	—	E	G to E	E
ADHESION TO FABRICS .. .. .	E	G	G	F	G	—	E	G to E	G
TEAR RESISTANCE .. .. .	G	F	G	P	F	P	G	F	F
ABRASION RESISTANCE .. .. .	E	G to E	G	P	G	P	E	G	E
COMPRESSION SET .. .. .	G	G	F	P	G	F	F to G	VG	F
REBOUND:									
Cold .. .. .	E	G	B	F	G	E	VG	G	G
Hot .. .. .	E	G	VG	F	G	E	VG	E	G
DIELECTRIC STRENGTH .. .. .	E	E	E	F	P	G	G	G	E
ELECTRICAL INSULATION .. ..	G to E	G to E	G to E	F to G	P	E	F to G	F to G	G
PERMEABILITY TO GASES .. ..	F	F	VL	L	F	F	L	VL	L to VL
ACID RESISTANCE:									
Dilute .. .. .	F to G	F to G	E	F	G	E	E	E	E
Concentrated .. .. .	F to G	F to G	E	F	G	F	G	E	VG
SOLVENT RESISTANCE:									
Aliphatic hydrocarbons .. ..	P	P	P	E	E	P	G	E	G
Aromatic hydrocarbons .. ..	P	P	P	G	G	P	F	E	F
Oxygenated (ketones, etc.) ..	G	G	G	G	P	F	P	P	P
Lacquer solvents .. .. .	P	P	P	G	F	P	P	P	P
RESISTANCE TO:									
Swelling in lubricating oil ..	P	P	P	E	VG	F	G	E	G to E
Oil and gasoline .. .. .	P	P	P	E	E	F	G	E	G
Animal and vegetable oils ..	P to G	P to G	P	E	E	F	G	E	G
Water absorption .. .. .	VG	G to VG	VG	F	F to G	G	G	VG	VG
Oxidation .. .. .	G	G	E	G	G	E	E	E	E
Ozone .. .. .	F	F	E	E	F	E	E	O	O
Slight ageing .. .. .	P	P	VG	G	P	E	VG	VG	O
Heat ageing .. .. .	G	VG	E	F	E	O	E	O	E
Flame .. .. .	P	P	P	P	P	F	G	G	E
Heat .. .. .	G	E	E	P	E	E	E	E	E
Cold .. .. .	E	E	G	F	G	E	G	G	G

E—Excellent G—Good F—Fair P—Poor L—Low O—Outstanding V—Very B—Bad

[Courtesy: Du Pont Co. Ltd.]

Table 2. Chemical resistance to inorganic salts and alkalis at room temperature

	N.R. or SBR, soft	N.R. or SBR, ebonite	Nitrile, soft	Nitrile, ebonite	Butyl	Neoprene	Hypalon
Aluminium chloride, aluminium sulphate, alums, ammonia 25%, ammonium chloride, ammonium persulphate, ammonium sulphate, barium sulphide, copper cyanide (in solution with alkali cyanides), copper sulphate, magnesium sulphate, magnesium chloride 50%, potassium (or sodium)-acid sulphate, -antimonate, -chloride, -cyanide, -sulphide, -thiosulphate, potassium cuprocyanide, tin chloride (stannous or stannic), zinc chloride, zinc sulphate .. .. .	S	S	S	S	S	S	S
Ammonium hydroxide, potassium (or sodium) sulphite ..	S	S	S	S	S	L	S
Calcium bisulphite, copper chloride .. .. .	L	S	S	S	S	S	S
Calcium chloride, ferrous sulphate (copperas) .. ..	S	S	S	S	S	S	S
Calcium hypochlorite .. .. .	S	S	L	S	S	L	S
Ferric chloride, nickel acetate, potassium (or sodium) bisulphite .. .. .	L	S	S	S	S	L	S
Potassium (or sodium) hydroxide .. .. .	S	S	L	S	S	S	S
Potassium (or sodium) hypochlorite .. .. .	S	S	S	S	L	L	S
Silver nitrate .. .. .	L	S	L	S	S	S	—

S—Satisfactory service X—Not generally recommended L—Limited range of applications

Table 3. Chemical resistance to acids at room temperatures

	N.R. or SBR, soft	N.R. or SBR, ebonite	Nitrile, soft	Nitrile, ebonite	Butyl	Neoprene	Hypalon
Acetic .. .. .	X	S	L	S	L	S	S
Acetic anhydride .. .. .	X	S	L	L	L	L	S
Aqua regia, nitric fuming .. .. .	X	X	X	X	X	X	X
Arsenic, benzoic, boric, carbonic, citric, fluoboric, fluosilicic, gallic, hydrocyanic, maleic 10%, salicylic 15%, tannic, tartaric .. .. .	S	S	S	S	S	S	S
Chlorine water .. .. .	S	S	S	S	S	X	S
Chromic 50% .. .. .	X	X	X	X	X	X	L
Formic .. .. .	X	S	S	S	S	L	S
Hydrobromic .. .. .	S	S	L	S	S	S	S
Hydrochloric, sulphuric 50% .. .. .	S	S	L	S	S	L	S
Hydrofluoric .. .. .	S	S	L	L	S	L	S
Lactic, sulphurous .. .. .	X	S	S	S	S	S	S
Malic .. .. .	S	S	S	S	S	S	—
Nitric 8% .. .. .	X	X	X	X	S	X	S
Nitric 20% .. .. .	X	X	X	X	L	X	S
Nitric 40% .. .. .	X	X	X	X	X	X	S
Oxalic 25%, 50% .. .. .	L	S	S	S	S	L	S
Phosphoric 80% .. .. .	S	S	L	S	—	S	S
Picric .. .. .	S	S	L	—	L	L	S
Sulphuric 20%, 25% .. .. .	S	S	L	S	S	S	S

S—Satisfactory service can be expected X—Not generally recommended L—May be suitable in a limited range of applications  
No entry means that insufficient information is at present available

ageing resistance is not as good as that of neoprene. Butyl rubber, on the other hand, has good weather resistance and excellent ageing resistance. Because of its very low gas permeability it is frequently used for gas-liquid phases where its high temperature resistance is an added advantage. Butyl rubber is resistant to dilute nitric acid and is being increasingly employed where resistance to animal and vegetable oils and fatty acids is required. A newer synthetic rubber is chlorosulphonated polyethylene (*Hypalon*), which has proved to be resistant to oxidising acids such as chromic acid (40%), phosphoric acid (85%), sulphuric acid (93%) and nitric acid (20%) as well as hydrogen peroxide (50%).

An even more recently developed rubber is co-polymer of vinylidene fluoride and hexafluoropropylene (*Viton*), which can be used for resistance to extremely corrosive chemicals, fuels and solvents. Laboratory tests have shown that *Viton* resists attack from concentrated nitric acid, hot aqueous hydrochloric acid, hot sulphuric and aqueous hydrofluoric acids. In Tables 2 to 4, the chemical resistance of some commonly used rubber linings is given. It must be remembered that good chemical resistance in any one particular rubber depends on the details of compounding; by varying the proportion of reinforcing fillers, accelerators, plasticisers and antioxidants as well as the rate of cure, both the physical and chemical prop-

erties of the lining can be substantially altered over a wide range. Much also depends upon actual service conditions. For example, a lining that can withstand an acid or an alkali separately might break down if exposed to frequent alternations of the two. The presence of ingredients in the liquor in trace quantities may have a cumulative effect on the lining.

#### Chemical resistance of ebonites

Ebonites generally have somewhat greater chemical resistance than the corresponding soft rubbers. Natural ebonites and those made from SB-R are satisfactory for use with demineralised water, acetic, formic, lactic and sulphurous acids where soft rubbers would not be recommended. Nitrile



ebonite can be used with nitro-benzene where a soft nitrile rubber would swell or weaken. An added advantage of ebonite is that it can be polished to a smooth finish; this is important in the food industry. Also pumps, valves and cocks can be easily machined to close tolerances after vulcanisation.

Nowadays, it must be noted that the distinction between soft rubber and ebonite is by no means as clear-cut as it used to be. New techniques have been evolved for making flexible ebonites by incorporating a proportion of neoprene or butyl rubber into a natural or SB-R ebonite stock. These rubbers, which do not form true ebonites themselves, act as plasticisers to give a much tougher and less brittle ebonite than would be possible with natural rubber or SB-R alone.

#### Temperature resistance

Most rubbers and ebonites begin to soften between 60° and 70°C.; they are generally not recommended for continuous use above 65°C. Special compounds made from SB-R can be used up to 80°C., but they tend to become brittle with age. Nitrile ebonites can resist temperatures up to 110°C., but their main drawback is that they are difficult to process.

Butyl rubber may be used up to

90°C. and in special compounds up to 105°C. Neoprene has the same temperature range; it is rarely used above 100°C. For extremely high temperatures, *Viton* is practically the only suitable rubber available. It resists temperatures up to 225°C. in continuous service and up to 315°C. in intermittent service. Because of its easy processability, it is possible to obtain high heat and fluid resistance in close tolerance components such as precision seals, O-rings and coated fabric. (It is claimed that *Viton*-coated fabric is equivalent to stainless steel in fireproofing qualities.) Unfortunately, it is rather expensive compared with the cost of neoprene.

#### Applications

Before the second world war, ebonite was widely used in the chemical industry for plant items such as pumps, propellers, casings, etc. Nowadays the large number of plastics have almost completely displaced ebonite as a material of construction for chemical plant. The only extensive application of both rubber and ebonites are as plant linings; so far, plastics have not managed to encroach on this field to any appreciable extent.

The use of rubber and ebonite linings as a protection against corrosion

has been a normal part of industrial practice since the first world war. It is important to consider the bond between rubber lining and metal as a true chemical bond since any attempt to pull the two apart will normally result in the rubber tearing before the bond yields. For this reason, rubber linings are suitable for use either under vacuum conditions or in pressure vessels where any sudden release of high pressure would cause any lining that was merely applied with adhesives to peel or blister. As important as applying the lining is the choice of vessel to be lined. Steel is usually preferred on account of strength, low cost and ease of fabrication. However, other metals may be used, such as cast iron, aluminium and brass—even wooden vats are sometimes lined. It is preferable that vessels are of welded construction; riveting is not desirable. However, if riveting cannot be avoided, the rivets should be flat and countersunk flush with the adjacent surface with no undercutting or crevices round the rivet heads. All sharp corners and angles in the vessel should preferably be avoided and particular care is required where vessels have internal strengthening ribs, baffles or other projections. If necessary, filler welds should be used on corners and

Table 4. Chemical resistance to organic materials at room temperatures

	N.R. or SBR, soft	N.R. or SBR, ebonite	Nitrile, soft	Nitrile, ebonite	Butyl	Neoprene	Hypalon
Acetone .. .. .	S	S	X	S	S	X	X
Alcohols .. .. .	S	S	S	S	S	L	S
Amyl alcohol, beet-sugar liquors, butyl alcohol, casein, detergents, ethyl alcohol, ethylene glycol, glucose, methyl alcohol, propyl alcohol, soap solution, sugar, triethanol- amine .. .. .	S	S	S	S	S	S	S
Aniline hydrochloride .. .. .	X	S	S	S	L	X	X
Aniline oil .. .. .	L	S	L	L	L	X	X
Beer .. .. .	S	S	X	X	S	S	S
Bleach liquor .. .. .	L	S	S	S	S	L	S
Buttermilk .. .. .	X	S	X	X	X	S	X
Castor oil, hydroquinone .. .. .	L	S	S	S	S	S	S
Catsup, coconut oil, cottonseed oil, linseed oil .. .. .	X	S	S	S	S	S	S
Cresol .. .. .	X	X	X	X	X	X	X
Ethers .. .. .	X	X	S	X	L	L	S
Ethyl acetate, methyl chloride .. .. .	X	X	X	X	L	X	X
Fatty acids .. .. .	X	L	L	L	L	L	L
Formaldehyde .. .. .	L	S	S	S	L	L	S
Fruit juices .. .. .	S	S	S	S	L	L	S
Furfural .. .. .	X	S	X	L	—	L	L
Glues, glycerine .. .. .	S	S	S	—	S	S	S
Hexane .. .. .	X	X	S	S	X	S	S
Mineral oils .. .. .	X	L	S	S	X	L	S
Nitrobenzene .. .. .	X	X	X	S	L	X	X
Olive oil .. .. .	X	L	S	S	S	L	S
Petroleum oils .. .. .	X	L	S	S	X	L	L
Phenol .. .. .	L	S	X	S	X	X	X
Sweet milk .. .. .	X	S	—	—	—	—	—
Turpentine .. .. .	X	X	S	S	X	X	X
Vegetable oils .. .. .	X	L	S	S	S	L	—
Wines .. .. .	S	S	S	—	L	L	S
Zeolites .. .. .	S	S	S	S	S	S	—

S—Satisfactory service X—Not generally recommended L—Limited range of applications

(Tables 2, 3 and 4 courtesy: Plant Lining Group, Federation of British Rubber and Allied Manufacturers)

all angles smoothed to a radius of not less than  $\frac{1}{4}$  in.

If castings are to be used, they must be close-grained grey iron, free from porosity or surface blowholes. All surfaces, whether steel or cast iron, must be free from weld spaths or pits. They should be left free from grease and unpainted and, above all, should not be galvanised. In the last instance, zinc can be removed by shot-blasting, but there is always the possibility of some traces remaining, with a consequent reduction in bond strength. It is much easier to line closed tanks if they are constructed with a detachable flanged end giving easier access for cleaning and lining and for the circulation of steam or hot air in vulcanisation. Thus vessels can often be constructed in two or more sections which can be separately lined and then assembled on site. This helps subsequently during inspection, cleaning and any repairs that may be required (including stripping and re-lining).

#### Application of lining

The process of applying a rubber lining, though simple in principle, is a highly skilled job. The vessel must first be thoroughly cleaned and degreased, usually by sweating in steam and shot-blasting. Bonding agents (usually isocyanates) are then sprayed or painted on and the unvulcanised sheets of calendered rubber (usually from  $\frac{1}{8}$  to  $\frac{1}{4}$  in. thick) are laid on and carefully rolled down by hand over the whole area of contact. Special precautions must be taken at the seams between one sheet and another and round all angles and corners.

It is essential that no air be trapped under the rubber, otherwise the bond will be weakened. The rubber sheet can be pressed into close contact with the metal using heated iron tools to force out the air. At the seams the heat and pressure weld the sheets into a homogeneous lining and, before vulcanising, the quality of the workmanship is tested by a dry spark test. If there is any flaw in the lining a spark is obtained by applying a high potential at 20,000 to 25,000 V. over the whole surface. The smallest fault is then revealed by a clear bright spark.

Vulcanisation is carried out in an autoclave, using saturated steam or hot

air at temperatures between 110° and 153°C. and pressures of 40 to 60 p.s.i. Components that are too large to fit into an autoclave can be lined on site by introducing hot air, hot water or low-pressure steam into the vessel itself. After vulcanisation, the lining is once again tested by means of an electric spark test. A rubber-lined tank is shown in Fig. 5.

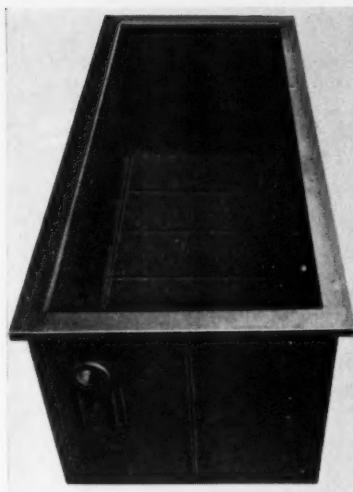
#### Pipes and other plant

Application of lining to pipework is as important, and fraught with difficulties, as lining of tanks. Seamless tubing is usually preferred and the tubing should be manufactured in accordance with B.S. 806:1954, Class C. Where it is impractical to use seamless material, tubing in accordance with B.S. 1387:1957, Class Medium, may be used. The maximum length of pipe which can be conveniently lined depends on the bore. For 3 in. bore and less, a maximum length of 9 ft. is advisable—for larger bores, lengths of up to 20 ft. can be accommodated. The smaller tubing is lined by drawing a tube of the uncured compound through its length attaching the flange coverings and vulcanising under pressure. The largest sizes are sufficiently large to have hand-rolled linings. Pipes for lining as well as bends, tees and crosses should have flanged or plain ends and allowance should be made for the lining thickness when specifying the bore (see Fig. 6).

Large stirrers, fans, centrifuge baskets, etc., are covered with unvulcanised sheet rolled on carefully by hand and cured in a suitably sized autoclave. Pumps and valves can also be lined with either soft rubber or ebonite. In smaller sizes, however, fabricated ebonite pumps, valves and pipes are available and, provided they are adequately supported and not required to work at high temperatures, they have a remarkably long life (see Fig. 7).

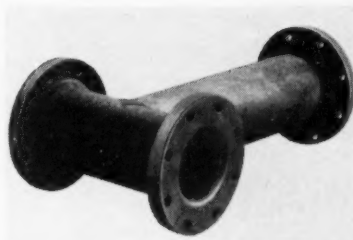
#### Uses

The amount of industrial equipment lined or covered with rubber or ebonite is steadily increasing. Examples of lining can be found in all branches of the chemical industry. Fig. 8 shows a typical example of a neoprene-lined



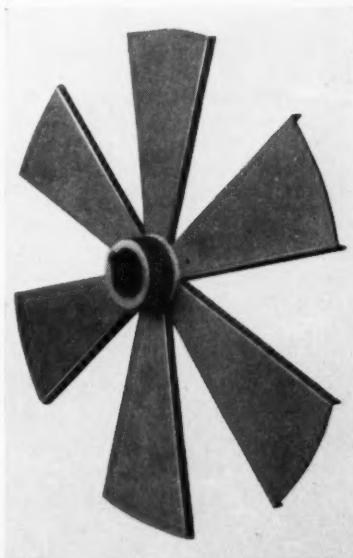
[Courtesy: B.T.R. Industries Ltd.]

Fig. 5. Rubber-lined tank



[Courtesy: B.T.R. Industries Ltd.]

Fig. 6. Pipe tee-section lined with rubber



[Courtesy: B.T.R. Industries Ltd.]

Fig. 7. Propeller vane lined with rubber

The material of construction for chemical plant to be discussed in next month's issue of **CHEMICAL & PROCESS ENGINEERING** will be

**ZINC COATINGS**

Table 5

Cost per sq. ft. lining, $\frac{1}{8}$ in. thick, for open-topped tank		
	s.	d.
Natural rubber or ebonite ..	9	0
Butyl rubber ..	10	9
Neoprene ..	13	0
Nitrile rubber ..	15	0

tank used by gold mining companies in the Orange Free State. The tanks, 8 ft. long and 50 ft. in diameter, are used to clarify pregnant solutions from solids consisting mainly of siliceous matter. The pregnant solution passes directly to the ion-exchange columns from which uranium oxide is extracted. Other examples of neoprene-lined tanks are for use in vats for washing abrasives. In one case, a sulphuric acid solution bath is heated to 80°C. by the injection of live steam. The abrasive particles are agitated by steam pressure with four separate batches of abrasives being washed every 24 hr. A 4-mm.-thick neoprene lining was used in these abrasive washing tanks to replace a 15-mm. coating of lead used previously, which failed after only six months' service.

Other uses for neoprene lining are in steel and concrete vats used in pigment manufacture, where neoprene is the only lining material to withstand the sudden temperature changes from boiling to freezing, which are necessary in this operation.

Another example is a 200-cu.-ft.-capacity vat for recovered hydrochloric acid which was lined with neoprene sheet over a thin base of polyisobutylene. Polyisobutylene prevents hydrochloric acid from being diffused through the neoprene to the surface of the tank. The neoprene successfully resists traces of the organic solvent in the vat.

An example of *Hypalon* lining is in

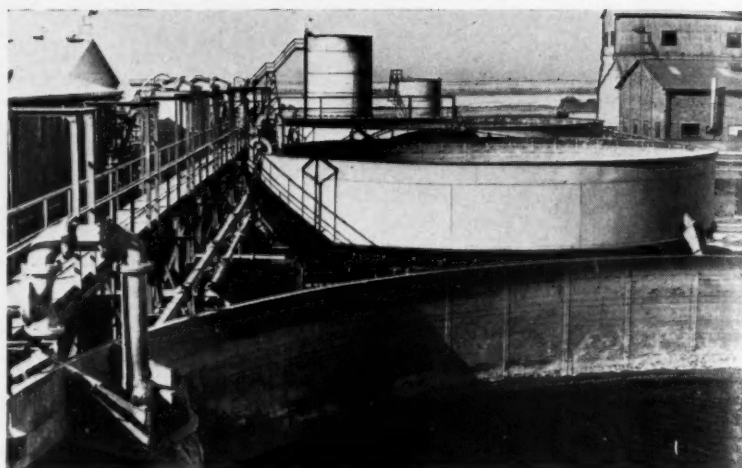


Fig. 8. Neoprene-lined tank used to clarify pregnant solutions from solids at a South African uranium plant

a chemical process where 75% phosphoric acid is concentrated and by-products recovered from the operation. A *Hypalon*-lined entrainment separator receives entrained liquid phosphoric acid, gaseous hydrofluosilic acid and steam from a vessel at 250° to 275°F. This lining has been in continuous operation since 1957. Fig. 1 shows the interior of a vat for processing uranium, titanium, vanadium and cobalt, lined with neoprene. Examples involving natural rubber lining include rubber-lined wash towers and piping for a chlorine absorption plant. Fig. 9 shows part of a battery of rubber-lined stock tanks for the storage of hydrochloric acid. Each stock tank is 30 ft. long and 7 ft. 6 in. diam.

#### Cost of lining

It is not simple to state the cost of plant linings. Much depends on the shape and size of the plant, the condition of the surface, the type of cure

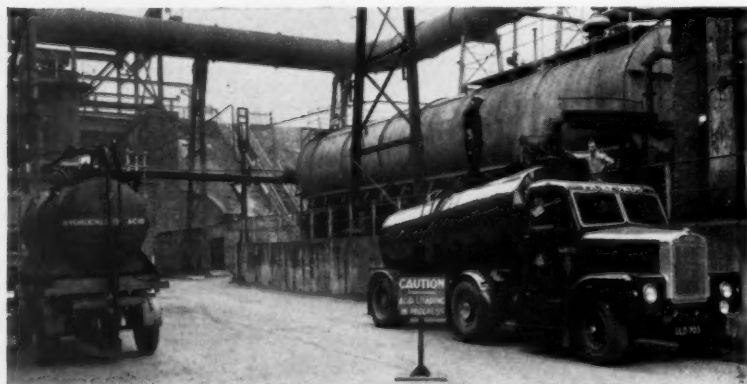
involved and the thickness of the rubber lining. In order to give some idea of the cost range involved, a standard case is quoted for an open-topped cylindrical or rectangular tank with an area of not less than 200 sq.ft. Vulcanisation is carried out by means of saturated steam. It is assumed that the surface is in fairly good condition. For normal duty a rubber lining of  $\frac{1}{8}$  in. is sufficient; if a highly abrasion-resistant lining is required, extra thickness must be used because the rubber compound will contain a higher proportion of rubber and less carbon black, i.e. a gum stock. Table 5 gives the costs for various rubbers. It may be added that the cost of lining a mild steel tank with rubber or neoprene is considerably less than fabricating the tank in stainless steel (whose corrosion resistance is not always as good).

#### Conclusion

It is evident from the foregoing that the main application of rubbers and ebonites as materials of construction for chemical plant is in the form of linings for tanks, vats, piping, stirrers and other plant units. The fabrication of plant from ebonite has been superseded by the use of phenolic resins, polystyrene or reinforced plastics. With the development of synthetic rubbers having increased chemical resistance and very high temperature resistance (such as *Viton* and *Hypalon*), but which are still relatively expensive, the application of a thin rubber lining onto a mild steel plant unit will become increasingly useful and important in the chemical industry.

#### REFERENCE

- <sup>1</sup>Rubber in Chemical Engineering', Natural Rubber Development Board, 1951.



[Courtesy: B.T.K. Industries Ltd.]

Fig. 9. Stock tank and rubber-lined piping for handling hydrochloric acid at Magnesium Elektron Ltd., Manchester



## CPE DIARY

**NOVEMBER 13 TO 14 Twenty-fifth Anniversary congress and commemorating exhibition** of the Society of Chemical Engineers, Japan, to be held in Kyoto. Details from the Institution of Chemical Engineers, 16 Belgrave Square, London, S.W.1.

**NOVEMBER 13 Annual general meeting** of the Royal Institute of Chemistry to be held at the University of Leeds.

**NOVEMBER 13 TO 18 Second Materials and Design Exhibition and Conference** to be held at Earls Court, London. Sponsored by Industrial & Trade Fairs Ltd.

**NOVEMBER 27 TO DECEMBER 1 Twenty-eighth Exposition of Chemical Industries** to be held at the New York Coliseum. Details from Mr. E. K. Stevens, 480 Lexington Avenue, New York 17.

**NOVEMBER 29 TO 30 Conference on oil hydraulic power transmission and control** to be given by the Hydraulic Plant and Machinery Group of the Institution of Mechanical Engineers. Details from Secretary, 1 Birdcage Walk, London, S.W.1.

**DECEMBER 7 TO 8 Symposium on sintered high-temperature compounds** to be held at the Hoare Memorial Hall, Church House, London, S.W.1.

### DECEMBER ISSUE OF CPE

Some articles in next month's issue of particular interest to our readers are:

Principles of Corrosion Applied to Chemical Plant

By H. A. Holden

Corrosion-resistant Epoxide Linings

By D. A. Shingleton

Northern Ireland's Chemical Industry

by I. L. Hepner

CPE Unit Operations Review—Centrifuging

By T. A. Barker

Materials of Construction for Chemical Plant—Zinc Coatings

By A. D. MacLennan

## Personal Paragraphs

★ Whiffen & Sons Ltd. have recently appointed **Mr. A. Robinson** as chairman of the board of directors, **Mr. A. H. Pashley** as vice-chairman and **Mr. A. P. Lowes** and **Mr. A. S. Woodhams** as joint managing directors.

★ A.E.I. Ltd. have appointed a director of research. He is **Mr. L. J. Davies**, who was formerly director of research of A.E.I. (Rugby) Ltd. **Dr. J. E. Stanworth** succeeds Mr. Davies at Rugby.

★ The George Cohen 600 Group Ltd. announce that they have appointed **Mr. P. Bonner** to the board of their associate company, T. C. Jones & Co. Ltd.

The retirement is also announced of **Mr. C. E. G. Nye** from the board of George Cohen Sons & Co. Ltd.

★ **Dr. F. C. Lloyd** has joined F. W. Berk & Co. Ltd. in the position of development director. Dr. Lloyd was formerly research manager of B.X. Plastics Ltd.



Mr. D. Y. Smith



Mr. L. E. Puddefoot

★ **Mr. P. H. Bohart** has been appointed senior vice-president of the Gulf Oil Corp. and will be responsible for the executive supervision of the crude oil department.

★ **Mr. Godfrey Davies** has become assistant managing director of Humphreys & Glasgow Ltd. **Mr. R. Withers** and **Mr. D. Lennon** both join the board of directors and **Mr. D. Yates** is appointed an associate director and head of Humphreys & Glasgow's construction department.

★ The prize of 25 guineas for student members, instituted in 1960, has been given for the first time to **Mr. J. P. Catchpole**, of the Department of Chemical Engineering, University of Birmingham, for his paper entitled 'Frequency Response Techniques for the Study of Packed Bed Characteristics'.



Mr. P. T. Fletcher



Dr. F. C. Lloyd

★ **Mr. H. Foxwell Sherborne**, managing director of Yorkshire Imperial Metals Ltd., has been appointed the new president of the British Non-Ferrous Metals Federation in succession to **Mr. W. W. Dolton**, managing director of the Delta Group of companies.

★ **Mr. P. T. Fletcher** has been appointed to the board of directors of United Power Co. Ltd. and will be responsible for construction and supply. Mr. Fletcher was previously deputy managing director at the U.K.A.E.A.'s Development and Engineering Group at Risley and he was, since April, special adviser to the Member for Reactors.

★ Geigy (Holdings) Ltd. announce that **Viscount Rochdale**, who is a member of their board, will be succeeding **Mr. A. H. Whitaker** as chairman of their subsidiary company, James Anderson & Co. (Colours) Ltd.

★ **Mr. A. D. Thompson** has been appointed deputy managing director of Edwin Danks & Co. (Oldbury) Ltd. and **Mr. C. H. G. Hayward** and **Mr. H. Harding Smith** have been appointed directors on the board of that company.

★ The Polytechnic Institute of Brooklyn, U.S.A., has created a new rank in recognition of meritorious service and accomplishment, to be known as Distinguished Professor. The first recipient of this rank is **Donald T. Othmer**, now Distinguished Professor of Chemical Engineering at Brooklyn Polytechnic.

★ **Mr. L. E. Puddefoot** has been appointed director of supply of B.B. Chemical Co. Ltd., Leicester. He has been technical director since 1944.

★ Brooks Instrument Ltd. have now established a company in England with **Mr. D. Y. Smith** as managing director.

# CPE Company News

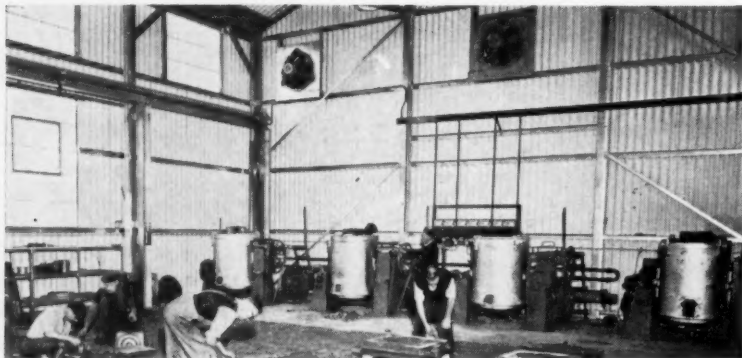
## New foundry

Kestner Evaporator & Engineering Co. Ltd. have inaugurated their new foundry at Greenhithe. This is the first part of the complete move of their works from New Cross to Greenhithe. Other shops and plants will later be transferred.

The new foundry is making castings of *Tantiron* (the company's acid-resisting silicon iron) and lead-alloy-resisting castings. Five furnaces have been installed, giving an output of 10 tons/week of *Tantiron* castings. There is room to increase this plant to double the output.

## Light-wall and duct fittings factory

Wilmot Breeden Ltd. have recently acquired a factory at Birmingham in order to launch an extensive manufacturing programme for *Truflo* stain-



The new factory for 'Tantiron' of Kestner Evaporator & Engineering Co. Ltd.

less-steel light-wall fittings for the petroleum, chemical and nuclear processing industries and for the manufacture of *Velflo* circular duct fittings for high-velocity air conditioning systems.

## Coated titanium anodes

I.C.I. Ltd., Magneto-Chemie N.V., Schiedam, Holland, and the Amalgamated Curacao Patents Co. of Curacao have agreed to pool their appropriate patents and collaborate to develop and extend the use of titanium anodes coated with an extremely thin film of platinum or similar metal. These offer very considerable advantages over conventional materials in industrial electrolytic cells for chemical manufacture, electroplating plant and equipment for converting brine to drinking water.

## Polypropylene film factory

Agreement has been reached in principle between the Metal Box Co. Ltd. and Shorko (a joint venture between Shell and National Distillers & Chemical Corp., New York) for the formation of a joint company in the U.K. It is intended that the joint company shall, in the first instance, erect and operate a factory in the U.K. for the manufacture of polypropylene film and film products.

## Polyethylene film plant

Plans to double the capacity of the Du Pont Co.'s polyethylene film plant at Richmond, Virginia, have been announced. When the expanded facilities are in full operation, the plant will be able to produce more than 60 million lb. p.a. of packaging film.

Construction will begin immediately

and manufacturing facilities of the Dispersed Pigments Division of Acheson Industries (Europe) Ltd. Cabot Corp. of Boston will also acquire the business and manufacturing facilities of the Acheson Dispersed Pigments Co., Philadelphia, and its subsidiaries and affiliates. It is expected that the transactions will be completed by the end of the year.

## New valve company

Serck Ltd. and the Jamesbury Corp., Massachusetts, have formed a new company, Jamesbury-Serck Ltd., Gloucester, to manufacture and market the Jamesbury double-seal ball valve. The new company will operate on a world-wide basis and undertake the development, manufacture and marketing of this valve and operating equipment. Manufacture will first be developed in England, but subsequently in a number of countries.

## Plastic pipe society

The Plastic Pipe Manufacturers Society has recently been formed and 16 of the major companies in the industry have become members. The chairman of the new society is Mr. F. H. A. Mackrill.

The main objects of the society are to encourage product research and to ensure the highest standards of quality in all plastic piping offered by the member companies. Members are committed to adhere to the requirements laid down in any British Standard for plastic pipe and will work towards the general adoption of the 'kite' mark of the British Standards Institution.

## Heat exchanger agreement

Birwelco Ltd. have made an exclusive agreement with Yuba Consolidated Industries Inc., Oklahoma, to manufacture and sell *Aimco Transaire* air-cooled heat exchangers in Europe and the British Commonwealth.

These are designed for the oil refining, petrochemicals and chemical processing industries, and are particularly useful in areas where the water supply is inadequate or unreliable.

## European headquarters

Monsanto Chemical Co., U.S.A., have announced the establishment of a European headquarters in Geneva by the company's wholly owned subsidiary, Monsanto Overseas S.A.

This move consolidates a number of company operations that have been in existence at several locations in Europe and is partly due to the success of Monsanto Research S.A. of Zurich.

and the additional facilities are expected to start production by about the middle of 1962.

## Steelworks automation company

The A.E.I./Davy-United Steelworks Automation Unit, which was formed in July 1960 to apply automation to the processing of steel and non-ferrous metals, will henceforth trade in its own right.

The interests of A.E.I. Ltd. and Davy-Ashmore Ltd. in this field are now combined in a jointly owned company, Davy-A.E.I. Automation Ltd.

## Sales-purchase agreement

Acheson Industries (Europe) Ltd. and Cabot Carbon Ltd. have concluded a sale-purchase agreement under the terms of which Cabot Carbon Ltd. will purchase the business

# What's New



## in Plant • Equipment • Materials • Processes

CPE reference numbers are appended to all items appearing in these pages to make it easy for readers to obtain quickly, and free of charge, full details of any equipment, machinery, materials, processes, etc., in which they are interested. Simply fill in the top postcard attached, giving the appropriate reference number(s), and post it.

### Sucrose detector

An instrument to detect the presence of dissolved sugar in water is to be marketed by William Bobby & Co. Ltd.

The device involves the use of three simple reagents and an automatic built-in heater to boil the sample. The presence of sugar in the sample produces a colour change which is detected photo-electrically. The prototype model was originally based on the Bobby *Hardicon*, a residual water hardness detector, and the final detection is carried out in the same way.

With the new detector, the presence of as little as 2 p.p.m. of sugar can be detected. The equipment can be fitted with an alarm relay to operate an alarm signal, or alternatively a shut-down or dump valve. **CPE 1751**

### Catalytic reforming plant

A catalytic reforming plant for the conversion of refinery petroleum gases into standard town gas has recently come on stream in Australia. This new Onia-Gegi plant, installed and constructed by Humphreys & Glasgow Ltd. at the works of Geelong Gas Co., Victoria, consists of two catalytic reforming units, each with a capacity of 1.5 million cu.ft./day of town gas. The feed stock will be tail gases piped five miles, direct from the Shell refinery. Alternative feed stock will be liquefied petroleum gases.

Refinery gases mixed with steam pass through a nickel-base catalyst at a temperature of 950°C. approx. The hydrocarbon steam reaction takes place to yield a reformed gas having a calorific value of 350 B.T.U./cu.ft. approximately. This is further enriched with additional refinery gas. **CPE 1752**

### Pneumatic controls

Colt Ventilation Ltd. are marketing their pneumatically-operated ventilators. One to 30 ventilators of all types can be controlled from one valve.

Black nylon or copper tubing connects the control valve to the pneumatic ram and the tubing can be taken round obstacles and yet, it is claimed, remain completely static after installation. The equipment is designed to operate on any supply pressure within the range 80 to 150 p.s.i. An overriding fusible link can be incorporated in the control assembly, ensuring the automatic opening of the ventilator in event of fire. **CPE 1753**

### Compressed air equipment

A compressed-air filter incorporating an automatic drain device and a new airline lubricator has been developed by Doulton Industrial Porcelains Ltd.

The new autodrain filter operates on the same principles as the F. 101 Doulton compressed air filter. In addition to a multi-stage cleaning action, it has an extended body which accommodates the float portion and pilot valve of the automatic drain mechanism. The main scavenger valve remains open for a short time after the pilot valve has closed, to permit complete drainage of the sump. Provision for manual draining is made.

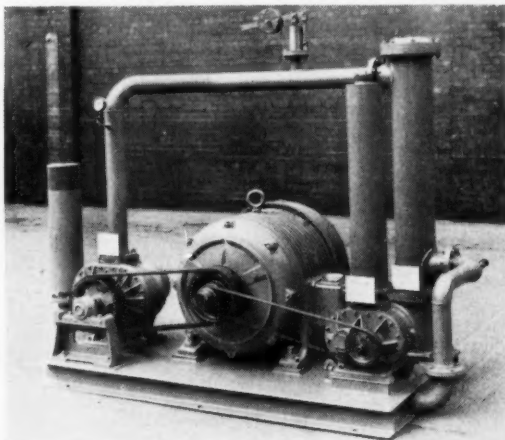
The air line lubricator can be adjusted over wide limits to determine the amount of oil mist introduced into the compressed-air system, and gives visual indication of the oiling rate. Atomisation at air flow rates of less than 1 cu.ft./min. is said to be ensured.

This compressed-air equipment is provided with line connections screwed  $\frac{3}{8}$  in. B.S. **CPE 1754**

### Valve positioner

The Fisher type 3560v/p valve positioner is now being manufactured by the Fisher Governor Co. Ltd. This instrument, which is designed to replace the type 3500, is said to combine high accuracy, speed and sensitivity, with low air consumption and ease of adjustment. Its break frequency at 5% of full instrument pressure range has been improved from 0.15 to 3.9 c/s. This overcomes the disadvantage of using a diaphragm actuator in a process control loop. Where a typical time lag may have been 1 sec. or more, the type 3560v/p has reduced this to 0.03 sec. Interchangeable characterised cams are used to enable the control valve characteristic to be more closely matched to the process

A Holmes-Connersville positive air blower, type 'RBS' tandem unit, capable of supplying oil-free air at pressures up to 20 p.s.i.g. and used by Nabisco Foods Ltd. The unit consists of a 7 in. by 8 in. and a 5 in. by 8 in. 'RBS' blower driven by a 65 h.p. motor and delivering 440 s.c.f.m. at 20 p.s.i.g. It is used for the pneumatic conveying of flour from storage hoppers at a rate of 600 lb./min. to weighing machinery **CPE 1755**





characteristic, helping to solve instability problems caused by an oversized control valve. **CPE 1756**

### PTFE extrusions

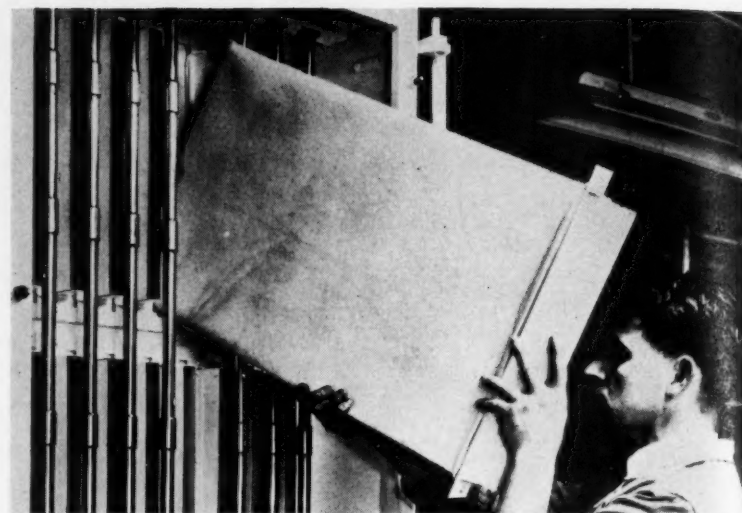
Crane Packing Ltd. have developed solid raw material forms of PTFE in a number of grades. Grades 7 and 8 have glass filament added to reinforce the basic polymer, thus giving added resistance to wear and deformation under load. They are suitable for use as bearing surfaces, such as bushes and thrust discs, and have been applied at maximum loadings of 40 p.s.i. at temperatures of up to 200°C. and at running speeds of 1,000 surface ft./min. under dry running conditions. They are supplied as mouldings and extrusions or as tubing of various wall thickness—or in component forms. **CPE 1757**

### Anti-corrosive coating

A new addition to the *Adcora* range of anti-corrosive coatings made by E. F. Richardson Ltd. is the *Adcora Pervon*. This is a polyurethane pitch coating which, it is claimed, cures rapidly at all temperatures. It can be applied effectively both to blast-cleaned metallic surfaces and to those prepared by less thorough methods; but for continuous immersion, blast cleaning and a dry film thickness of 10 to 15 mils is recommended. It is said to have a good resistance to ammonia, acetic acid, nitric acid, hydrogen peroxide, fatty acids and alkalis even after prolonged immersion. *Adcora Pervon* is also unaffected by boiling distilled water. **CPE 1758**

### Protection switch

A flow failure protection switch has been developed by Nash & Thompson



The Dallow Lambert Dalamatric filter showing main access door hinged open and the tiered assembly

Ltd. which is adaptable to different forms of control in high and low temperature or corrosive industrial processes. It can be fitted directly into piping systems from  $\frac{1}{2}$  to 36 in. diam. and consists of a screw-in venturi readily exchangeable for different rates of flow. This operates a system of metal bellows, and the movement of the liquid in the bellows is transmitted to an electrical circuit or through a differential transformer to various kinds of meter. In this way, the switch can be used either as an alarm device or as an indicator of changing rates of flow. **CPE 1759**

### Automatic fabric dust collector

Dallow Lambert Ltd. have introduced their new *Dalamatric* automatic fabric filter. Whilst employing limited quan-

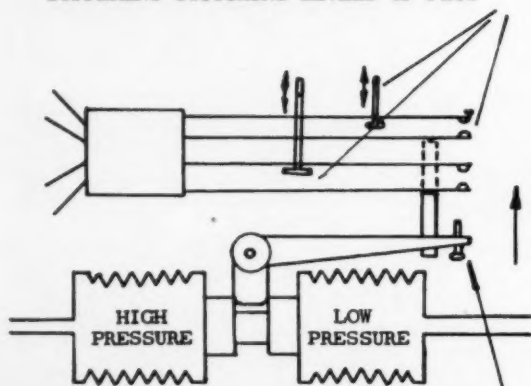
ties of compressed air as a cleaning medium, it embodies the fundamental dynamic air flow characteristics generally associated with some blow-ring-type filters, but has no mechanical moving parts. The design incorporates a system of standard filter units, so as to enable installations to be assembled capable of handling air volumes of 100,000 cu.ft./min. or more.

The *Dalamatric* may consist of one-, two- or three-tier assemblies. The maximum fabric area of a single three-tiered unit is 180 sq.ft. of fabric, but multi-assemblies will increase the filtration area. Dust initially retained on the filter is removed to the hopper below and discharged by conventional methods. **CPE 1760**

### Fire retardant

Bentonite, a fine clay used in the foundry, civil engineering, oil and animal foodstuffs industries, is also valuable as a fire retardant. When mixed with water, it will absorb up to six times its dry weight in water and expand 12 to 15 times its original dry volume. The water in the slurry and in the treated vegetation must be driven off as water vapour before the fuel can be heated to its ignition temperature. If the heat energy of an advancing fire front is insufficient to do this, the fire's spread will be stopped. The moist coating will remain effective for up to 2 to 3 hr. under severe American summer drying conditions. The slurry is relatively non-abrasive to pumps and is very much lighter than borate slurry. F. W. Berk & Co. Ltd. are the U.K. distributors of bentonite. **CPE 1761**

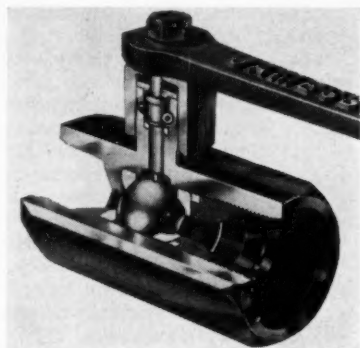
ADJUSTMENT FOR SETTING GAP TO GIVE DIFFERENT SWITCHING LEVELS OF FLOW



MECHANICAL OVER-TRAVEL ADJUSTMENT

## Side-entry mixers

A range of side-entry mixers has been designed by Premier Colloid Mills Ltd. for use in vessels which are too deep to accommodate top-entry mixers with long shafts. They can be used for petroleum blending, sludge control in storage tanks and blending of lubricating and crude oils, etc. The three units available can be used singly or in double or triple banks, thus making it possible to handle quantities from 15,000 to more than 1 million gal. The output shaft speed is 500 r.p.m. with motor sizes of up to 15 h.p. depending on the model. Totally enclosed or flameproof motors are available. The mixing heads are propellers, and the shaft is manu-



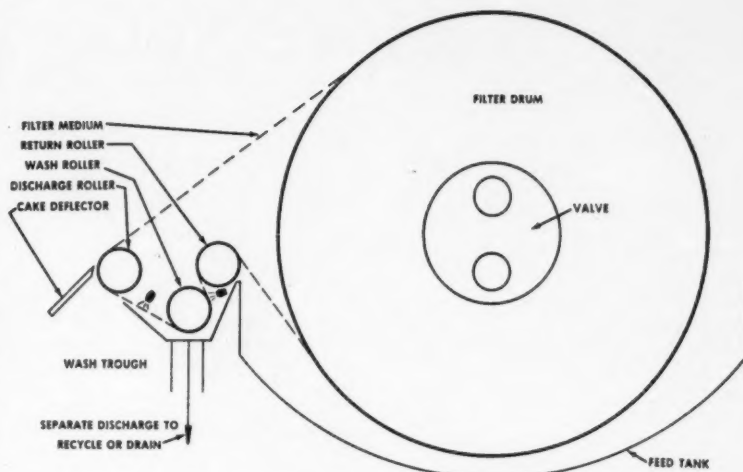
A Jamesbury double-seal ball valve, manufactured by the new company, Jamesbury-Serck Ltd., consists of a sphere rotating between two seats made in such materials as PTFE, nitrile rubber, neoprene, nylon, etc. A seal with a flexible lip gives an initial sealing force, independent of line pressure. **CPE 1762**

factured in lengths and materials to suit requirements. The largest model, No. 3800, can be mounted directly to the tank without support from the ground. A bevel drive brings the motor weight close to the mixing tank, thus keeping down the bending moment to a minimum. **CPE 1763**

## Peat for industry

A new factory has recently been opened by Alluvial Products (Ireland) Ltd. and F. W. Berk & Co. Ltd. for converting peat into a material known as *Berkool*. This is used in foundries in the manufacture of castings and is added to the mould mixture to reduce expansion defects. Sand mixtures containing peat also mould well and are not so liable to crumble as those containing materials such as wood flour.

The first factory opened by these two companies was at Birkenhead in 1958, Bord na Mona supplying the raw peat.



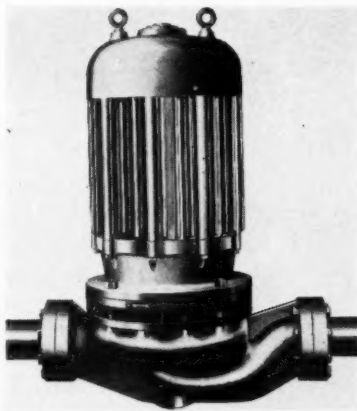
Diagrammatic layout of the 'EimcoBelt' filter. Clean continuous cake discharge can be achieved without the use of a scraper blade or air blow. The filter belt is removed from the drum and is washed before being returned. A zipper joint in the belt enables it to be changed quickly, and belt alignment is maintained automatically. Belts resistant to almost any form of chemical attack can be supplied. **CPE 1766**

The new plant will have a capacity of 12,000 tons of *Berkool*. **CPE 1764**

## Rotary acid pump

Kestner Evaporator & Engineering Co. Ltd. are marketing their tantiron *Rotopump* for transferring acids where a positive self-priming pump is needed. The *Rotopump*, a small positive rotary pump of the vane type and constructed throughout of tantiron, is resistant to most corrosive fluids, but is not recommended for use with hydrochloric or hydrofluoric acids.

Self-priming, light and portable, it is available in two sizes and as completely motorised units or as bare shaft pumps. It is especially suitable for handling strong sulphuric acid, but not for abrasive laden liquors. **CPE 1765**



The Sigmund WP-N in-line process pump

## Process pump

Sigmund Pumps Ltd. have introduced a new in-line, single-stage, single-entry centrifugal pump, WP-N. No concrete foundation or baseplates are required, and the close-coupled arrangement reduces the number of component parts required and improves ease of dismantling, re-assembling and alignment. By removing a few bolts the complete rotating element and motor can be lifted clear of the pump casing for inspection, without disturbing the pipework. A mechanical seal is fitted, and it is claimed that radial and axial thrust have been eliminated.

The pump can be made in a variety of materials to suit specific requirements. **CPE 1767**

## Powder containers

The Paper Group of the Metal Box Co. Ltd. have recently introduced a new range of 8-in.-diam. composite drums with tinplate ends. These were shown at the International Packaging Exhibition. The drum body can be manufactured in a variety of wall thicknesses out of waterproof cartridge paper, American Kraft liner or chipboard. The string-opening throw-away drum and the slip-lid type are both produced in heights from 2 to 20 in. The reclosure string-opening model is available from 3 to 20 in.

The drums are designed for packing chemical and pharmaceutical powders or pastes. **CPE 1768**



# Nuclear Notes

## Transplutonium samples

The U.S. Atomic Energy Commission will supply 12 samples of rare transplutonium elements to scientists in five European countries. The materials to be supplied are americium-243, curium-244, berkelium-249 and californium-252.

The countries to which the samples will go are Belgium, France, Western Germany, Norway and Sweden. All five countries have agreements for co-operation with the U.S. for research and development in the peaceful uses of atomic energy.

## A.E.C. changes

Major changes have been announced in the organisation of the operating staff of the A.E.C. The objectives of the changes are to improve the effectiveness of the organisation, shorten the chain of command and communication with field operations, strengthen the role of the operations office managers and relieve headquarters programme divisions of administrative and supervisory burdens.

## Particle accelerator

A proton beam accelerated to the design energy of 15 MeV has been obtained from the linear accelerator injector which forms the first stage of NIMROD, the 7,000-MeV proton synchrotron under construction at the Rutherford High Energy Laboratory of the National Institute for Research in Nuclear Science, Harwell.

Pulsed 15-MeV beams are delivered by the injector to the synchrotron where they are constrained to a circular path, and in this way they will make many revolutions until the output energy of 7,000 MeV is reached. The protons will then be directed to suitable targets for experiments in high-energy nuclear physics.

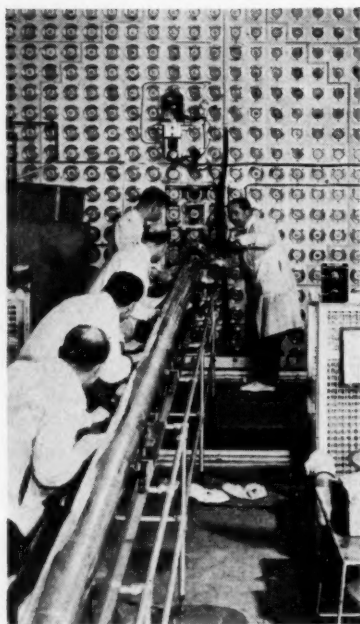
## Italian nuclear power station

The Garigliano nuclear power station being built by International General Electrical Operations S.A., a subsidiary of U.S. General Electric, for the Societa' ElettroNucleare Nazionale (S.E.N.N.), is scheduled to be completed in 1963. Its design is similar to that of the 180-MW Dresden nuclear power station and is of the dual-cycle type. It will provide 150-MW net electrical output to a distribution system in Southern Italy.

The principal components of the nuclear steam supply system have been designed to allow for the future installation of 50% additional plant output, provided this additional power is obtainable from the core. If the output is increased, the station may produce power almost competitive with power costs from conventional oil-fired plants. Initially it will cost about 10% more than power from an equivalent fossil-fuelled plant.

## Philippine nuclear prospects

A report recently published by the I.A.E.A. shows that a relatively large nuclear power plant installed late in the 1960s on the Island of Luzon in the Philippines might be economically competitive over its lifetime with a conventional power plant of the same size. There would be enough demand to justify the installation of a large thermal plant in the Luzon grid, and a nuclear power plant should be considered, bearing in mind that its costs would be less subject to fluctuation than oil prices for oil-fired plants.



A large tube being loaded into the core of the reactor BEPO (British Experimental Pile Zero Energy) at Harwell. This natural uranium-fuelled graphite-moderated reactor can attain a power of 6,000 kW and is cooled by air. The tube shown will be used to contain materials at high temperatures for research purposes

## Reactor dismantled

The *ad hoc* advisory committee appointed by the U.S. Atomic Energy Commission to consider the future use or disposition of the stationary low-power reactor No. 1 (SL-1) in Idaho, has concurred in proposals for decontamination of the reactor building and dismantling of the core. The SL-1 was the reactor involved in the accident that occurred on January 3, 1961, at the National Reactor Testing Station in Idaho.

## Gas-cooled reactor

The Federal German Minister for Atomic Energy, Prof. S. Balke, has allotted 18 million DM. towards the cost of planning, development and trials for a gas-cooled prototype reactor. The total costs for development work are estimated at 25 million DM. This project is part of a comprehensive programme to develop gas-cooled reactors, and the major function of this particular reactor will be to obtain experimental data of operational characteristics for this particular type. This work was suggested by the German Atomic Commission.

## Fuelling reactors

Experiments have been carried out in the Calder Hall reactors to find out what happens when fuel elements are loaded into a reactor on full power. The element experienced the required temperature changes and no significant effects on the elements as a result of on-load charging were revealed.

A second experiment was carried out to see what effects frequent variations in temperature—thermal cycling—had on fuel elements, and no measurable ill effects were found.

## Research computer

An ATLAS electronic digital computer has been ordered for the National Institute for Research in Nuclear Science. The computer, together with the necessary buildings, will cost about £3½ million. It will be installed at the Institute's Rutherford High Energy Laboratory, Harwell, for common use by the universities, the U.K.A.E.A., government departments and the N.I.R.N.S. itself. It should be ready for use early in 1964. The ATLAS computer, made by Messrs. Ferranti, has been developed in co-operation with scientists at the University of Manchester.



### Spectral shift reactor project

The Babcock & Wilcox Co. announced recently that they have been awarded a \$211,000 contract by the A.E.C. for a plant analysis study of their heavy/light-water-moderated reactor, known as the 'spectral shift' control reactor. A \$1.2-million A.E.C. contract for a basic physics study of the reactor was awarded to the company last year. Work will be carried out with the firm of Stone & Webster Engineering Corp. to make an economic analysis of the reactor system for the A.E.C.

The spectral shift reactor is expected to increase the life of water reactor cores and the efficiency of fuel consumption, and lower the installed cost of nuclear electric generating plants.

### I.A.E.A. symposium

Mr. J. Pelsler, technical director of Reactor Centrum Nederland, recently held a lecture on the use of the 10-kW ARGONAUT-type research reactor at Petten, during a symposium on 'Programming and Utilisation of Research Reactors'. The symposium was organised by the I.A.E.A. Mr. Pelsler recently sat on an international commission for the judgment of safety aspects of the research reactor BR-2 at Mol.

### Superheat reactor

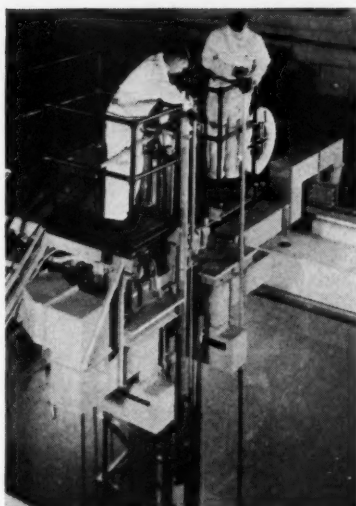
An \$8-million ESADA superheat reactor is now under construction near Pleasanton, California. This is a joint project of the Empire State Atomic Development Associates Inc. (ESADA) and General Electric and will be a flexible fuel test facility designed to provide information required for the development of reliable superheat fuel elements for the development of large, economic nuclear superheat power plants.

The ESADA reactor has been designed so that it can be later modified to operate as an integral superheat reactor.

### I.A.E.A. board of governors

Colombia, Greece, Hungary, Pakistan and Viet-Nam have been elected to serve a two-year term on the board of governors by the General Conference of the I.A.E.A. These new members succeed Bulgaria, Ceylon, Mexico, the Philippines and Spain.

The new membership of the board of governors for 1961-62 is thus: Argentina, Australia, Brazil, Canada, Colombia, Czechoslovakia, El Salvador, France, Federal Republic of Germany, Greece, Hungary, India,



The nuclear research reactor now in operation at Union Carbide's Research Centre, New York, is a pool-type reactor, designed and built by American Machine & Foundry Co., rated at 5,000 kW.

Iraq, Japan, Pakistan, Portugal, South Africa, Sweden, Thailand, U.S.S.R., U.K., U.S.A. and Viet-Nam.

### Laboratory reactor exhibited

An operating L-77 laboratory reactor, designed and built by Atomics International, was recently exhibited in Beirut, Lebanon, in the U.S. Atomic Energy Commission's Atoms for Peace programme. The L-77 is a low-cost reactor contained in a tank only 7 ft. high and 8 ft. in diam., small enough for school, industrial or laboratory purposes.

### Controlled nuclear fusion

A one-week international conference on 'Plasma Physics and Controlled Nuclear Fusion Research' was recently held by the I.A.E.A. at Salzburg. An almost complete up-to-date picture was obtained of the work in different parts of the world in the field of plasma physics aimed at achieving controlled nuclear fusion. It was decided to hold another conference on this subject in 1964.

### Nuclear fusion conference

A large international conference on plasma physics and controlled nuclear fusion research was recently held by the International Atomic Energy Agency (I.A.E.A.) at Salzburg. About 100 papers were presented to the conference.

In addition to further nuclear research as a whole, the Agency's conference is largely intended to help eliminate wasteful duplication of effort and expenditure. It will also show what progress has been achieved since the second U.N. International Conference on the Peaceful Uses of Atomic Energy, held in 1958, where classified results of fusion research were first made accessible to the scientific world at large.

### Fast reactor seminar

A seminar on the physics of fast and intermediate reactors was recently organised by the I.A.E.A. in Vienna. The programme included basic neutron physics data, integral experiments with critical assemblies, the methods of calculation and physics design of large systems, problems of breeding and fuel cycles, kinetics and the physics of the safety of fast reactors. Programmes of research and development in many countries were reviewed.

### Plasma physics conference

The I.A.E.A.'s conference on plasma physics and controlled nuclear fusion research recently took place at Salzburg.

This first international scientific meeting, devoted entirely to work in the field of plasma physics undertaken specifically for the achievement of controlled thermonuclear fusion, was attended by over 500 scientists from 28 countries and from seven international organisations. The results obtained by 476 research workers were submitted for discussion by the conference.

### French-Belgian Euratom project

A definitive contract for a 242,000-kW underground French-Belgian nuclear power plant has been awarded to Westinghouse Electric International Co. of the United States, Ateliers de Constructions Electriques de Charleroi of Belgium, and Framatome of France, by Societe d'Energie Nucleaire Franco-Belge des Ardennes (SENA).

The project for a closed-cycle water reactor of Westinghouse design was accepted by the Council of Ministers of Euratom under the programme of investment in large plants for the six-nation European Community.

### £ s d CHEMICAL PLANT COSTS

Cost indices for the month of September 1961 are as follows:

Plant Construction Index: 186.0

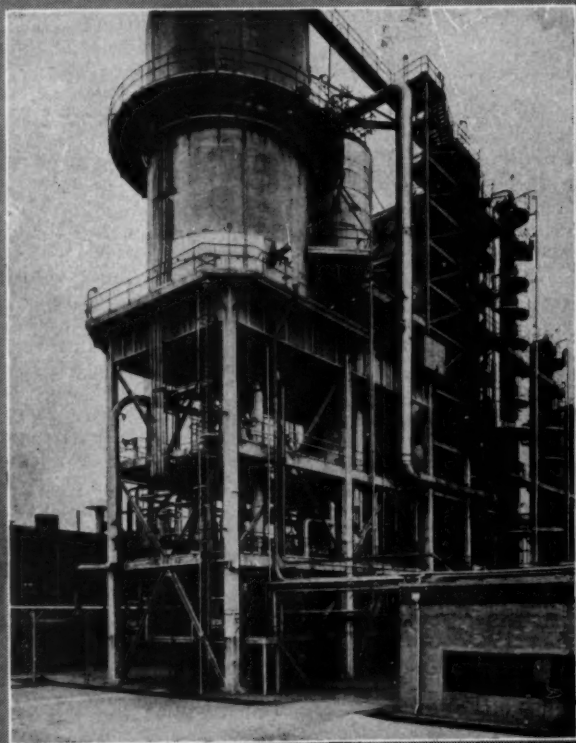
Equipment Cost Index: 174.9

(June 1949 = 100)

£ s d

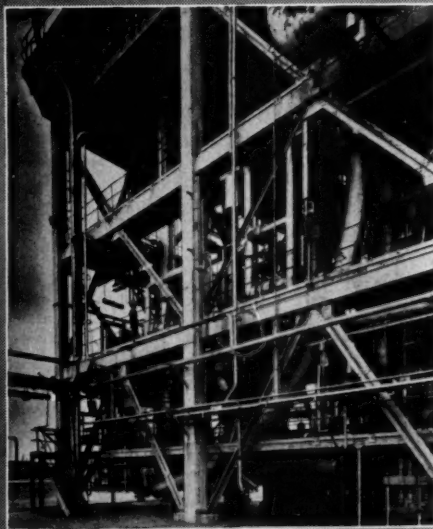
# PIPEWORK

## FOR PETROLEUM PLANT

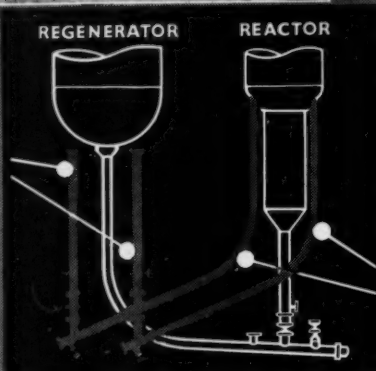


At the Grangemouth Refinery of B.P. Ltd., John Thompson (Pipework) Ltd., supplied the catalyst feed pipe lines from the regenerator, and the crude oil and catalyst mixture feed pipe lines to the reactor.

These lines which serve an integral part of the process have been supplied to B.P. Ltd., over a period of up to 10 years, and are completely fabricated to Class 1 specification of high grade mild steel for a temperature of 970°F at 30 lb/sq. at the Wolverhampton works of John Thompson (Pipework) Ltd.



Catalyst feed lines  
18 in. diameter.



Crude residual oil and catalyst mixture  
feed lines to reactor. 20 in. diameter.



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# New Books

**Atomic Energy Waste: Its Nature, Use and Disposal.** Edited by E. Glueckauf. Butterworths, 1961. 95s. net.

With the rapid developments which have taken place in the last decade in the building of nuclear power reactors, there has been increasing concern about safe disposal of radioactive waste products. It is the purpose of this book to describe the formation of such materials, the methods which are available for their treatment, their safe disposal, possible large-scale applications for some of the products as sources of radiation and the legal aspects of waste disposal.

Dr. Glueckauf, the editor, has arranged the book in six parts and has chosen acknowledged experts to contribute in their own special fields.

The first chapter discusses in detail the fission process and describes the growth and decay of fission products. There are many useful tables and illustrations. In the case of Fig. 18a-g, however, illustrating the elemental composition of fission products as a function of neutron flux and cooling time, the confusion of lines makes it difficult for the reader to use the graphs.

Part 2 describes the effects of radiation on materials and living organisms, the movement of radioactive substances in food chains and concludes with a brief statement of the problems of radiation protection. Following, in Part 3, the legal position with respect to atomic waste disposal is summarised and the main provisions of the Radioactive Substances Act, 1960, are outlined.

Part 4, which comprises a third of this book, is devoted to the operations involved in the treatment of radioactive waste. The various treatments available for low-active wastes are considered and the principal methods used in this country, in Canada and the United States are described. The problem of high-level active wastes is then examined and, whilst the principal of 'concentration and storage' must always hold, it is of interest to see how much research in this field is now directed towards conversion of large volumes of liquid to more concentrated solid waste. Although liquids undoubtedly present the greatest problem in waste disposal, the release of radioactive gases and vapours to the atmosphere is often necessary but must

be done under carefully controlled conditions and hence can present many difficulties in the safe operation of reactors, of 'hot' laboratories and of fuel reprocessing plants. An excellent short chapter discusses the dispersion of activity from chimney stacks and refers in particular to the nuclides argon-41, carbon-14 and iodine-131.

Parts 5 and 6 describe the uses for fission products as sources of radiation and the applications of such sources in agricultural research, food preservation and for inducing industrially important chemical reactions. These sections are well written and give good summaries of the present position with regard to the applications for radiation. Unfortunately, at present, with the exception of fuel element cooling ponds, only electrical machines and cobalt-60 are being used for industrial radiation purposes and the latter can scarcely be described as 'atomic waste' although it is produced in a nuclear reactor.

The book is very well produced and is a source of much useful information. It is, however, rather expensive. The chemical engineer will be most interested in Part 4 and will find the treatment in some of the other sections of this book, particularly in Parts 1 and 2, rather too detailed for his purposes.

G. R. HALL

**Plastics in Nuclear Engineering.** By James O. Turner. Reinhold Publishing Corp. Pp. 139. 44s. net.

The series of books of which this is the 20th volume so far, is aimed at a readership which includes 'students, engineers, designers and, in fact, all practical people in plastics', while the author claims that this volume is 'written from the viewpoint of the engineer who has the responsibility both of designing bevtrons and reactors, bubble chambers and perhaps

cyclotrons, and of making them work'. In spite of the expected experienced readership, the author has, as he explains, taken care to avoid history, chemistry and mathematics. Frankly, it seems more likely that the book was written very largely for the benefit of plastics manufacturers seeking further outlets for their products.

Be that as it may, one cannot dispute the fact that this little volume presents an admirable picture of the versatility of plastics. There is a most impressive array of applications of plastics in the nuclear field.

In the introductory chapter, the author deals with the classification of plastics, methods by which their properties can be changed and the devices for this purpose. It is a pity that radioactive decay and nuclear fission have been so badly mixed and ill-explained. There is (perhaps unavoidably) a tendency towards patchiness in certain details. For example, we are given an explanation of the role played by solvent, activator and shifter in a scintillation counter, but the mechanism of a BF<sub>3</sub> neutron counter is not explained. Chapter 2 deals with devices for radiation measurement, and Chapter 3 with radiation protection. There are chapters, too, on high- and low-temperature services, magnets, high vacua, optical devices and mechanical, thermal and miscellaneous uses. The final chapter deals with radiations.

It is rather striking that, although the moderator properties of plastics are mentioned, nothing is said of the use of plastics as moderators in small reactors, although this is a method adopted by certain American firms. Incidentally, every single application is taken from U.S. practice. It is surprising, too, to find no reference to the extensive use of plastics in the handling of such violently corrosive materials as fluorine, hydrogen fluoride and chlorine- and bromine-trifluoride, although all of these are used in nuclear processing.

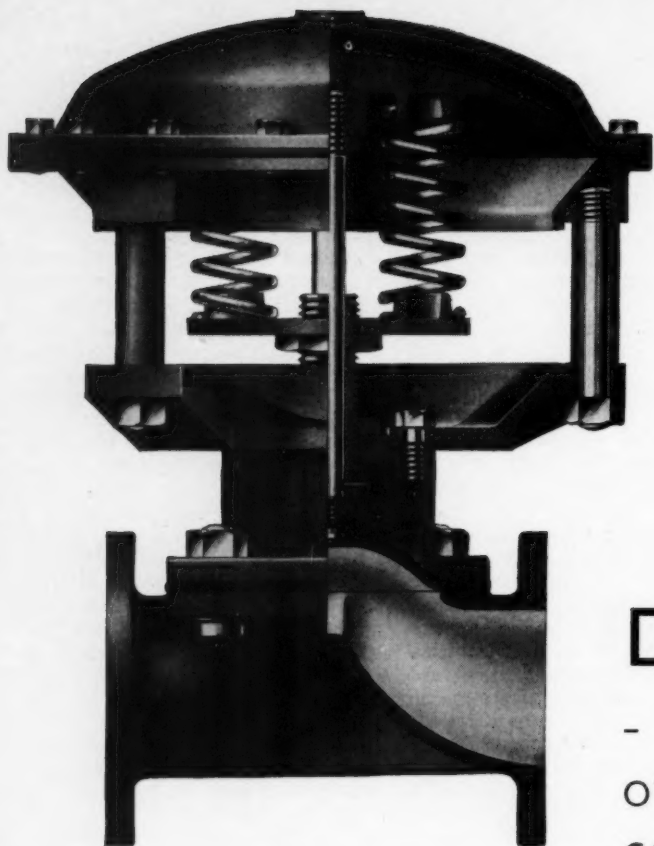
However, these are shortcomings which could quite easily be amended in a future edition; certainly there will be future editions, for this is a book which should be in considerable demand. It is generally well written, well arranged and contains an ample selection of photographic illustrations which are astonishingly good and very appropriate. This is a fascinating little volume, which cannot fail to appeal to students of the nuclear sciences as well as to plastics manufacturers and their sales promoters.

F. R. PAULSEN

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The snowball growth of chemical engineering is in no small measure due to the Saunders Valve. Many products and processes which are today commonplace were impracticable before the advent of Saunders Diaphragm closure and isolation.

The diaphragm makes sure closure and isolates operating gear from difficult and dangerous fluids.



# Orders and Contracts

## Water-treatment plant

The water treatment of the new power station at Basra, in Iraq, has been awarded to a British firm, the Neckar Water Softener Co. Ltd. Pre-treatment equipment to deal with 15,000 gal./hr. of river water has been installed, and the plant for second-stage treatment deals with 7,500 gal./hr. of the pre-treated total.

## Conveyor belting

Following negotiations during the recent British Trade Fair in Moscow, B.T.R. Industries Ltd. have been awarded an order worth over £250,000 for more than five miles of conveyor belting. Deliveries will commence in September.

This belting will be used for carrying iron ore and non-ferrous metals. It consists of an all-Terylene carcass with a nylon breaker and with specially compounded rubber covers for operating within a temperature range from 195° to -58°F.

## Nitrogen plant

A new plant which will increase the production capacity of nitrogen supplies to the Wilton Division of I.C.I. by 50% is to be built by British Oxygen.

Costing about £500,000, the plant will have a capacity of 5,000 cu.m./hr. of very high purity nitrogen (equal to about 140 tons/day) and will be installed at B.O.C.'s Middlesbrough works. The unit is expected to be running by the middle of 1962.

At the Middlesbrough works, two similar units are already supplying I.C.I. with nitrogen and a 200-ton/day tonnage oxygen plant serves local steelworks.

## Vacuum distillation columns

Constructors John Brown Ltd. have been awarded another contract, worth nearly £60,000, by Polimex, the Polish Government trade organisation. It is for the design and supply of two vacuum distillation columns incorporating Kittel plates. This equipment is to be used in a plant for the production of synthetic fibres, to be built in Poland.

## Gas reforming plant

A contract worth £300,000 for the design and installation of refinery gas reforming plant at Edinburgh has been

placed by the Scottish Gas Board with Humphreys & Glasgow Ltd.

The design will be of the *Onia-Gegi* self-steaming type, which produces sufficient waste heat steam for process use. The plant will comprise two units and produce a maximum output of 18 million cu.ft./day of 450 B.T.U. town gas. The plant will also be capable of reforming butane if required, and could be modified in future for light distillate reforming. It will be the first of the *Onia-Gegi* type to be built in Scotland.

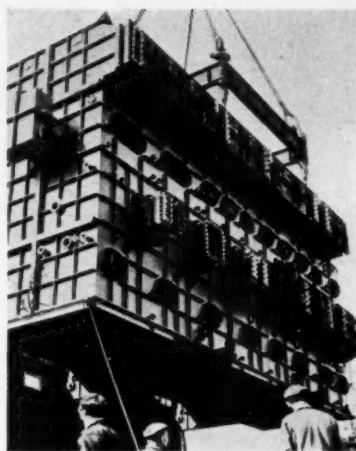
## Pulp plant agreement

Simon Handling Engineers Ltd. have negotiated an agreement with International Pulp Products Inc. for the supply of pulp mills for the paper industry.

International Pulp Products Inc. are the holders of the patents for the *Cusi* process for the use of bagasse, the crushed residue of cane sugar mills, straw and similar annual plants for pulp making. A plant using the process is successfully operating in Mexico, producing bleached and unbleached pulp.

## Research department

Diversey (U.K.) Ltd. and its associate company, Deosan Ltd., announce that the Diversey-Deosan chemical and bacteriological laboratories have now been amalgamated to form one joint research and development department. The company produces and



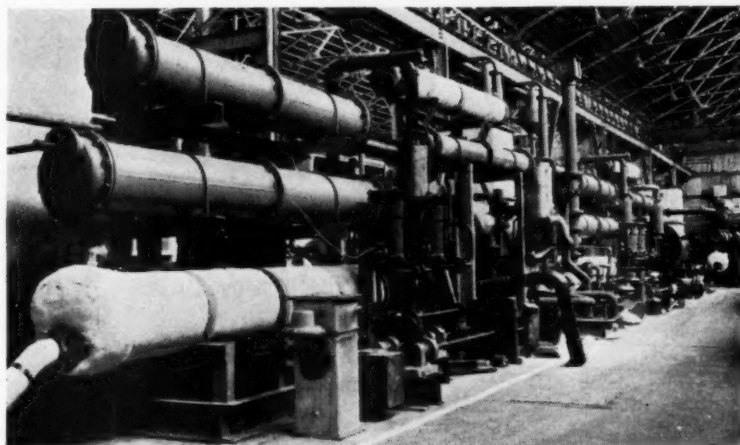
A marine Multiflash plant being installed in the new Shaw Savill liner Northern Star. Designed by G. & J. Weir Ltd., the unit employs 20 flash chambers and will produce 500 tons of fresh water/24 hr.

markets specialised sterilisers and detergents for the agricultural, food-processing, catering and metals industries.

## Electric arc furnaces

Steel, Peech & Tozer, a branch of the United Steel Co. Ltd., are to undertake a £10-million scheme for the replacement of all existing open-hearth steel melting furnaces by six 110-ton oxygen-lanced electric-arc furnaces, special attention being given to the fume-extraction equipment.

An order has been placed with W. C. Holmes & Co. Ltd. to supply gas cleaning plant for each of the first two furnaces to be installed at Templeborough works.



Hydrogen/nitrogen plants in course of assembly in the Smethwick works of Gas Atmospheres Division of the Incandescent Heat Co. They are for despatch overseas. The Division has orders for plant to be installed in Belgium, France, Poland, Russia, Portugal, Tanganyika, India and other countries

**U.S.S.R.****Beryllium deposits**

The rare beryllium mineral, melphante, has been discovered in Uzbekistan, Central Asia. Up to now this mineral has only been found in Norway and the Kola peninsula.

The mineral in Uzbekistan contains beryllium, calcium, magnesium, aluminum, silicon and sodium.

**NETHERLANDS****Metallic stearates plant**

The N.V. Koninklijke Stearine Kaarsenfabrieken 'Gouda-Apollo' have announced the formation of a subsidiary company, named Witco-Gouda Stearaten N.V., for the installation of a plant to produce a range of metallic stearates under the licence of the Witco Chemical Co. Inc., New York. The Witco-Gouda plant, under construction on the site of Gouda-Apollo at Gouda, will be put on stream within a few months, and the fatty acids required will be supplied by Gouda-Apollo.

**KUWAIT****Electrodialysis plant**

The Government of Kuwait has awarded a \$418,000 contract to Ionics Inc., Mass., for a 240,000-gal./day electrodialysis plant. This contract includes full cost of a four-month acceptance test and supervision of operation for a year subsequent to acceptance.

The plant will process underground water containing approximately one-tenth the salinity of sea-water from

Sulibeyah well fields to produce water for domestic and maybe for experimental agricultural purposes at a cost of approximately \$1/1,000 U.S. gal. The project is scheduled for completion in June 1962.

**HUNGARY****Argon**

Production of argon has been carried out at Pet since last autumn using tail gases from ammonia synthesis gases. Argon is obtained mixed with methane gas and is used for welding purposes after the methane has been removed. Yearly production of argon is about 160,000 cu.m. having a purity of 99%.

**GERMANY****Methanol**

Production of methanol during 1960 was increased by 36,000 to 330,000 tons. The increasing consumption of methanol which has been noted in Germany since 1959 has continued during 1960. The German price of methanol has, however, remained steady.

**YUGOSLAVIA****Research reactor**

A 100-kW. TRIGA Mark II research and training reactor and the special nuclear fuel required for its operation will be supplied to Yugoslavia under agreements between the I.A.E.A., the United States and Yugoslavia. The reactor is to be operated by the Jozef Stefan Nuclear Institute at Ljubljana. The transfer of the uranium is taking place under the agreement for co-operation concluded

between the agency and the United States in May 1959.

**SPAIN****Fungicide company**

A jointly owned Spanish company to manufacture and sell dithiocarbamate fungicides will be formed by Energia e Industrias Aragonesas S.A. and E.I. du Pont de Nemours & Co., U.S.A., subject to approval by the Spanish government. The new company, Desarrollo Quimico Industrial S.A., will initially produce *Manzate* maneb fungicide and *Parzate* zineb fungicide, both developed by Du Pont, and will build a plant at Sabinanigo, adjacent to an existing plant operated by Energia e Industrias Aragonesas.

**CANADA****Superphosphate plant**

The Electric Reduction Co. of Canada Ltd.—a member of the Albright & Wilson Group—have started production at their new \$40-million plant at Port Maitland, Ontario. Production consists of normal and triple superphosphate, phosphoric acid and liquid phosphatics for use in fertilisers and dicalcium phosphates for animal food.

Up to 500,000 tons of phosphate rock will be transported by rail to the site each year. Dicalcium phosphate, through competing with defluorinated phosphate rock and Curacao rock, provides the most available form of inorganic phosphorus for livestock food and should account for a major portion of the market.



Dicalcium phosphate being conveyed to the kiln at the Port Maitland, Ontario, plant of the Electric Reduction Co. of Canada Ltd.



